September 1955 Pages 489-568

The Journal of the SOCIETY OF DYERS AND COLOURISTS

Volume 71 Number 9

CONTENTS

PROCEEDINGS OF THE SOCIETY—

EXAMINATION FOR THE ASSOCIATESHIP (A.S.D.C.) HELD IN JUNE 1955 489

THE GUILDS OF THE CITY OF LONDON WITH SPECIAL REFERENCE TO THE WORSHIPFUL COMPANY OF DYERS (*P. E. Jones*) 496

THE DYEING OF TERYLENE POLYESTER FIBRE WITH DISPERSE DYES ABOVE 100°C. (*A. S. Fern*) 502

WEAR PROPERTIES OF RESIN-FINISHED RAYON STAPLE FABRICS—A NEW METHOD OF LABORATORY ASSESSMENT (*L. F. H. Brems and T. H. Morton*) 513

COMMUNICATIONS

BLEACHING JUTE WITH HYDROGEN PEROXIDE (*H. Chatterjee and K. B. Pal*) 525

AN EXPLANATION OF DYEING MECHANISMS IN TERMS OF NON-POLAR BONDING (*A. N. Derbyshire and R. H. Peters*) 530

THE IODINATION OF WOOL (*H. R. Richards and J. B. Speakman*) 537

NOTES 544

OBITUARY NOTICE 545

NEW BOOKS AND PUBLICATIONS 546

MANUFACTURERS' PUBLICATIONS AND PATTERN CARDS 549

ABSTRACTS 551

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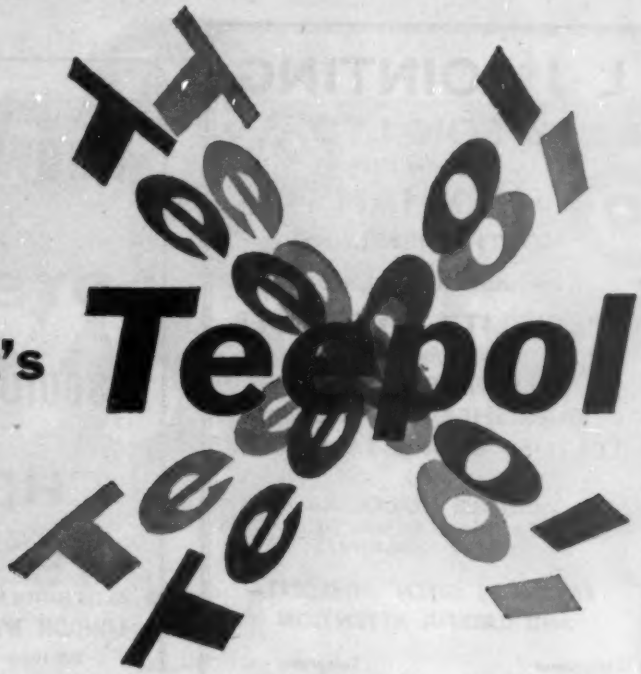

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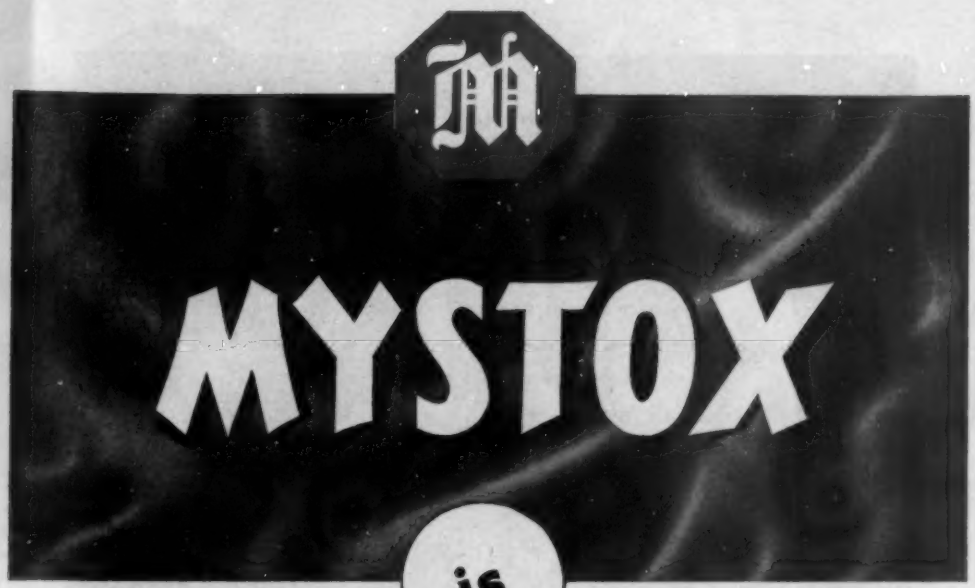

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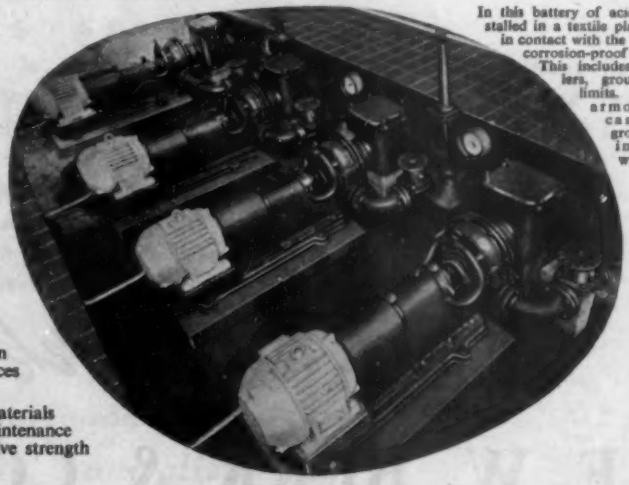

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1955 and pages 369–373 of the July 1955 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138–9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal* —

LECTURES

The Chemistry of Leuco Vat Dye Esters	*S. Ainsworth and A. Johnson*
Cellulose Acetate and Water Repellency	*R. J. Mann*
Printing of Wool including the Production of Mélange Effects	*J. A. Potter*
Estimation of Fluorescent Brightening Agents	*G. G. Taylor*
The Principles and Practice of Flameproofing Textiles	*F. Ward*
The Use of Silicones in the Textile Industry	*T. W. Watson*

COMMUNICATIONS

The Rotproofing of Viscose Rayon I — Treatment of Viscose Rayon with Formaldehyde and Formaldehyde-containing Resins	*J. W. Bell, M. M. Ramsey, and C. S. Whewell*
The Polarography of Azo Dyes	*J. de O. Cabral and H. A. Turner*
The Adsorption of Some Organic Vapours by Wool and Nylon	*H. R. Chipalkatti, V. B. Chipalkatti, and C. H. Giles*
The Irgalan Dyes — Neutral-drawing Metal-complex Dyes	*G. Schetty*

F.T.C.C. PUBLICATION

An Investigation into Some Methods for the Assessment of the Weather Fastness of Dyed Textiles	*E. Butterworth and J. C. Guthrie*

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FORTHCOMING MEETINGS OF THE SOCIETY

Friday 23rd September 1955

MANCHESTER SECTION. *The Causes of Anomalous Fading in Light Fastness Testing.* K. McLaren, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Textile Institute, 10 Blackfriars Street, **Manchester 10.** 6.30 p.m.

Tuesday, 27th September 1955

HUDDERSFIELD SECTION. Visit to Messrs. Samuel Fox & Co. 7 p.m.

SCOTTISH SECTION. *High Temperature Dyeing of Cellulosic and Synthetic Fibres.* Dr. A. Schmitz (Bayer Co.). St. Enoch's Hotel, **Glasgow.** 7 p.m.

Wednesday, 5th October 1955

MIDLANDS SECTION. *The Dyeing of Orlon and Orlon Mixtures.* I. M. S. Walls, Esq., B.Sc., Ph.D. (Imperial Chemical Industries Ltd.). Kings Head Hotel, **Loughborough.** 7 p.m.

Friday, 7th October 1955

LONDON SECTION. *The Contribution of Petroleum Chemicals to the Textile Industry.* H. Bratherton, Esq. (Shell Petroleum Co. Ltd.). The Royal Society Rooms, Burlington House, Piccadilly, **London, W.1.** 6 p.m.

BRADFORD JUNIOR BRANCH. *Dyeing Theory in Retrospect.* Dr. J. Simons (I.C.I. Ltd.). Technical College, **Bradford.** 7.15 p.m.

Tuesday, 11th October 1955

SCOTTISH SECTION. *Textile Research—The Problems of Different Fibres.* H. Corteen, Esq., M.Sc., F.T.I., F.Inst.P. St. Enoch's Hotel, **Glasgow.** Joint Meeting with the Textile Institute. 7 p.m.

Wednesday 12th October 1955

NORTHERN IRELAND SECTION. *The Dyeing of the newer Synthetic Fibres.* Dr. I. M. S. Walls (Imperial Chemical Industries Ltd.). Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

Friday, 14th October 1955

WEST RIDING SECTION. Supper and Smoker. Victoria Hotel, **Bradford.**

Tuesday, 18th October 1955

HUDDERSFIELD SECTION. *Dyeing Mixtures of Wool with 'Terylene' or 'Orlon'.* J. G. Graham, Esq. (Imperial Chemical Industries Ltd.). Co-operative Society Cafe, **Huddersfield.** 7.30 p.m.

Friday, 21st October 1955

MANCHESTER SECTION. *The Printing of Acetate Rayon Fabric.* W. B. Miller, Esq., M.Sc. The Textile Institute, 10 Blackfriars Street, **Manchester 10.** 6.30 p.m.

Monday, 24th October 1955

BRADFORD JUNIOR BRANCH. *Fading — Its Nature and Assessment.* H. B. Mann, Esq., M.Sc. (Courtaulds Ltd.). Technical College, **Bradford.** 7.15 p.m.

Tuesday, 25th October 1955

SCOTTISH SECTION. *Kier Boiling.* C. Garrett, Esq., F.T.A. St. Enoch's Hotel, **Glasgow.** 7 p.m.

Wednesday, 26th October 1955

MIDLANDS SECTION. *Textile Bleaching with Peroxide and Peracetic Acid.* L. Chesner, Esq., B.Sc., A.T.I., and G. C. Woodford, Esq. College of Technology, **Leicester.** 7 p.m.

Thursday, 27th October 1955

NORTHERN IRELAND SECTION. *Fire Prevention Methods in the Textile Industry.* Film will be shown. J. R. Coulter, Esq. Thompson's Restaurant, Donegall Place, **Belfast.** Joint meeting with Textile Institute. 7.30 p.m.

WEST RIDING SECTION. *The Dyeing and Finishing of 'Courpleta'.* W. R. Beath, Esq., and J. S. Ward, Esq. (Courtaulds Ltd.). Victoria Hotel, **Bradford.** 7.30 p.m

Friday, 4th November 1955

LONDON SECTION. *Microscopical Investigations of Fibre Damage.* G. G. Taylor, Esq., and J. C. Brown, Esq. (Clayton Dyestuffs Co. Ltd.). Joint Meeting with the London Centre of the Guild of Dyers and Cleaners. The Royal Society Rooms, Burlington House, Piccadilly, **London, W.1.** 6.30 p.m.

Tuesday, 8th November 1955

BRADFORD JUNIOR BRANCH. *Microscopical Investigations of Fibre Damage.* G. G. Taylor, Esq., and J. C. Brown, Esq. (Clayton Dyestuffs Co. Ltd.). Technical College, **Bradford.** 7.15 p.m.

LONDON SECTION. *The Design and Production of Furnishing Fabrics.* H. Woodman, Esq., A.T.I. (Warner & Sons Ltd.). The Chemical Society, Burlington House, **London, W.1.** 7 p.m.

Wednesday, 9th November 1955

NORTHERN IRELAND SECTION. *Causes of Felting in Rayon Textiles.* H. W. Best-Gordon, Esq. (Courtaulds Ltd.). (Joint Meeting with the Textile Institute), Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

Saturday, 12th November 1955

MIDLANDS SECTION. Ladies' Evening—Dinner Dance. Welbeck Hotel, **Nottingham.**

Tuesday, 15th November 1955

HUDDERSFIELD SECTION. *The Problems of the Garment Dyer and Finisher.* B. Kramrisch, Esq. and F. Grainger, Esq. Co-operative Cafe, **Huddersfield.** 7.30 p.m.

Friday, 18th November 1955

HUDDERSFIELD SECTION. Annual Dinner. Co-operative Cafe, **Huddersfield.**

MANCHESTER SECTION. Ladies' Evening — Full details later.

Tuesday, 22nd November 1955

SCOTTISH SECTION. *The Dyeing and Finishing of 'Courpleta'.* W. R. Beath, Esq., and J. S. Ward, Esq. (Courtaulds Ltd.). St. Enoch's Hotel, **Glasgow.** 7 p.m.

Wednesday, 23rd November 1955

MIDLANDS SECTION. *Modern Methods of Finishing Open-width Knitted Fabrics.* Film Evening introduced by J. Dalglish, Esq. Technical College, **Nottingham.** 7 p.m.

BRADFORD JUNIOR BRANCH. *Properties of Terylene/Wool Blends in Yarn and Fabric Form.* B. I. Brooke, Esq. (I.C.I. 'Terylene' Council). Technical College, **Bradford.** 7.15 p.m.

Friday, 2nd December 1955

LONDON SECTION. *The Application and Evaluation of Optical Bleaching Agents.* G. R. Perdue, Esq. (British Launderers Research Association). The Royal Society Rooms, Burlington House, Piccadilly, **London, W.1.** 6 p.m.

Wednesday, 7th December 1955

MIDLANDS SECTION. *The Dyeing and Finishing of 'Courpleta' Knitted Fabrics.* S. N. Bradshaw, Esq. Bell Hotel, **Leicester.** 7 p.m.

Friday, 9th December 1955

MANCHESTER SECTION. *The Causes of Felting in Rayon Textiles.* H. W. Best-Gordon, Esq., F.C.I. The Textile Institute, 10 Blackfriars Street, **Manchester 10.** 6.30 p.m.

Tuesday, 13th December 1955

SCOTTISH SECTION. *Basic Mechanism in Jigger Dyeing of Woven Fabrics.* Dr. J. Ashworth (Courtaulds Ltd.). St. Enoch's Hotel, **Glasgow.** 7 p.m.

Wednesday, 14th December 1955

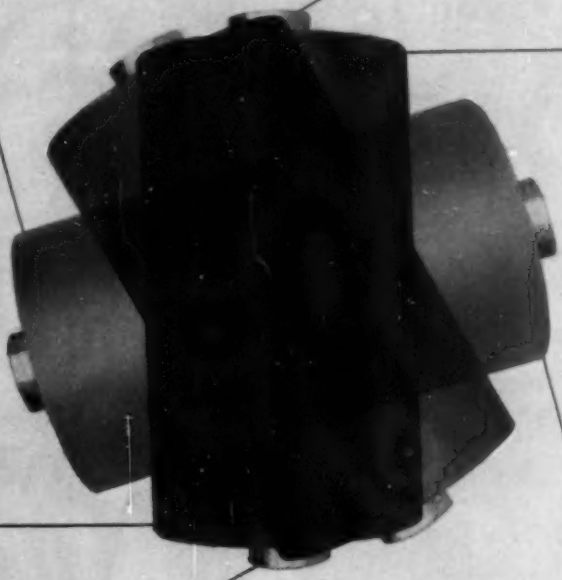
NORTHERN IRELAND SECTION. *Recent Developments in Bleaching, Dyeing and Finishing Machinery.* K. Laurie, Esq. (John Dalglish & Co. Ltd.). Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

Thursday, 15th December 1955

BRADFORD JUNIOR BRANCH. *Experience in the Application of Automatic Regulation to the Dyeing Process.* F. Blezzard, Esq. (Drayton Regulator & Instrument Co. Ltd.). Technical College, **Bradford.** 7.15 p.m.

continued on page xxxix

Colours for Nylon and other Synthetic Fibres



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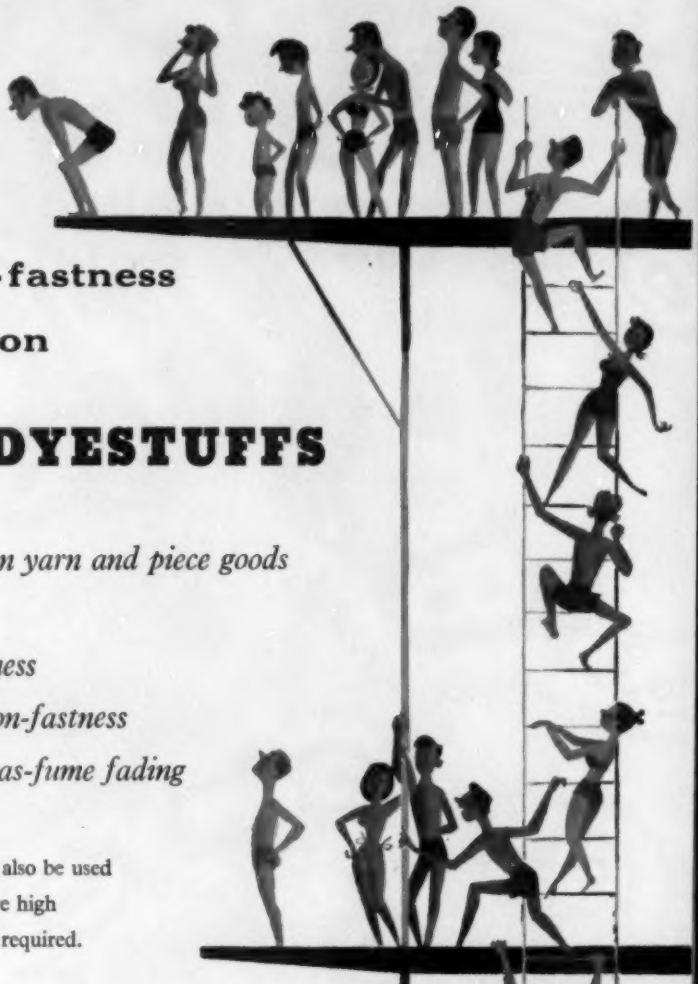

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THE JOURNAL

OF THE

Society of Dyers and Colourists

Volume 71 Number 9 SEPTEMBER 1955 *Issued Monthly*

Proceedings of the Society

Examination for the Associateship (A.S.D.C.) 1955

The second examination for the Associateship of the Society of Dyers and Colourists was held in June 1955, and below appear the examination papers and the report of the Diplomas Executive Subcommittee on the examinations. It is hoped that publication of these papers and the report will be helpful both to prospective candidates and to teachers, and that it will serve to inform members of the Society and others of the standard of knowledge expected from candidates for the Associateship.

Examination Papers

Papers A and B are taken by all candidates; but Papers C, D, and E are taken by a candidate only in his chosen branch of tinctorial technology. Further, in Paper E a candidate has a choice of subject—analytical and testing techniques or *the organisation of production and economics*

Paper A

General Chemistry and Properties of Dyes, Pigments, Auxiliary Products
General Chemistry and Properties of Organic High Polymers

10 a.m.–1 p.m. on Thursday, 9th June 1955

(QUESTION 1 must be attempted, together with TWO questions from Section I and THREE questions from Section II)

SECTION I

1. Compare the properties of soap with those of a secondary alkyl sulphate. Suggest additions which would improve the efficiency of either of these substances when it is used for scouring, and explain the reasons for this improvement.

2. Give an account of the general methods of preparation and the properties of **one** of the following groups—
(*a*) Triphenylmethane dyes
(*b*) Xanthen dyes
(*c*) Indigoid dyes.

3. Give an account of the chemistry of the essential stages in the formation of azo dyes from primary amines and coupling components. Name and give the constitution of (*a*) an azo dye for cellulose acetate **and** (*b*) an azo dye which forms a complex with a metal.

4. What is meant by a vat dye? Mention some relationships between the chemical structure and the fastness properties of anthraquinonoid vat dyes. State why care must be taken to keep the temperatures and the degree of alkalinity of vatting and dyeing of some of them low; give an example.

5. How is 2 : 3 - hydroxynaphthoic acid (β-hydroxynaphthoic acid) prepared and converted into products used for producing azoic dyeings and prints? In what respect are these products superior to 2-naphthol (β-naphthol) for this type of work, and how far can these points of superiority be related to the structural formulae of these compounds?

6. Describe with examples the preparation of a lake from a water-soluble dye, mentioning any points to which special attention must be paid.

7. State what atomic groupings must be present in dye molecules to confer the following properties on the dyes, illustrating your answer with examples—(*a*) high solubility in water, (*b*) capacity to form complexes with metals, **and** (*c*) ability to be diazotised and developed on the fibre.

SECTION II

8. Explain with specific examples what is meant by **three** of the following terms—
(*a*) Copolymerisation
(*b*) Cationic polymerisation
(*c*) Cross-linking
(*d*) Emulsion polymerisation.

9. Outline methods used in the study of the submicroscopic structure of high polymers, indicating the essential information obtained by each method.

10. Write concise notes on **three** of the following—

(*a*) Number-average molecular weight
(*b*) Intrinsic viscosity (limiting viscosity number)
(*c*) Second-order transition temperature
(*d*) Fractionation.

11. Write an account of the chemistry of **one** of the following types of high polymers—

(*a*) Polyamides
(*b*) Urea resins
(*c*) Silicones.

12. Show how modifications in the polymer chain can modify fibre-forming properties, mentioning particularly the effects of (*a*) chain length and (*b*) chemical nature of the monomeric unit.

13. Write a short essay on **one** of the following—

(*a*) Cellulose derivatives
(*b*) Ethenoid polymers.

Paper B

Theories of Colour
Relation of Colour to Constitution
Colour Assessment and Colour Fastness

2–5 p.m. on Thursday, 9th June 1955

(*SIX questions only to be attempted, including at least one question from each of the four sections of the paper*)

SECTION I

1. Describe in detail a colorimetric method of following the progress of absorption of disperse dyes by nylon yarn, emphasising the precautions you would take to ensure accurate results.

2. Employed in the testing laboratory of an impecunious dyeing firm, you are asked to undertake the estimation of the concentration of dye liquors by photoelectric methods. No colorimeter is installed, but two barrier-layer photoelectric cells and a sensitive galvanometer are available. Minor electrical and optical components such as resistances, lamps, and simple lenses can be purchased easily. Describe how you would construct a simple colorimeter with the minimum expenditure.

3. Colorimeters for absorption measurements are normally calibrated in terms of either "percentage transmission" or "optical density". Define these two quantities, and give the equations for the transformation of either into the other. What are the relative merits of the two quantities for the estimation of dye solutions? What are the laws of Beer and Lambert, and to what extent are they valid for dye solutions?

4. Describe the colorimetric principles involved in the analysis of solutions containing mixtures of two dyes. What are the conditions leading to optimum accuracy in such analyses? In certain cases two dyes may interact to form a complex. Describe how you would proceed to verify qualitatively the existence of a complex of this type. Is optical apparatus necessary for this purpose?

SECTION II

5. Explain and comment briefly on **four** of the following terms—

(*a*) Colour circle
(*b*) Standard observer
(*c*) Complementary dominant wavelength
(*d*) Extinction coefficient
(*e*) Illuminant C
(*f*) Equi-energy spectrum
(*g*) Brightness.

6. Write an essay on **one** of the following topics—

(*a*) The quantitative assessment of light fastness
(*b*) The evaluation of fluorescent brightening agents
(*c*) The uses of polarised light in the study of dyeing phenomena.

7. Why do certain dyed fabrics appear to change colour markedly when taken from artificial light into daylight, whilst other fabrics maintain a more constant colour? What methods have been used to assess the magnitude of these colour changes? What are the defects of these methods and how could they be improved?

8. You are in charge of a Utopian dyehouse which is equipped with a foolproof tricolorimeter by means of which it is possible to obtain rapid and highly accurate measurements of the reflectance of dyed material through arbitrary but permanent red, green, and blue colour filters. Would you use this instrument to promote the efficient working of the dyehouse; and if so, how? If you would not use the instrument, give your reasons.

SECTION III

9. Certain dyes are widely used as indicators in solutions of different pH. Indicate the structural features which are thought to be responsible for these effects, and explain how the change of colour is brought about.

10. Outline a modern theory of the relationship between colour and chemical constitution. Use it to explain the colour differences between the two basic dyes which are cations of *pp′p″*-tris-dimethylaminotriphenylmethane and *pp′*-bis-dimethylaminotriphenylmethane respectively.

11. What changes in the absorption spectrum of a dye solution might take place if the dye aggregated to form a colloidal suspension? How would you confirm that the changes were in fact due to aggregation? How would you attempt to decide whether the aggregates were amorphous or microcrystalline?

12. Write a short account of how the study of **either** (*a*) cyanine dyes **or** (*b*) carotenoid pigments has assisted the development of theories of the relationship between colour and chemical constitution.

SECTION IV

13. A certain dye A has the same absorption spectrum in aqueous solutions containing either 10% of pyridine or 10% of ethyl alcohol. Dye B behaves in the same way. A mixture of the two dyes, however, does not give the same absorption spectrum in the two solvents. Determine from the data below the composition of the mixture, showing how the more satisfactory data are selected.

	450 mμ.	550 mμ.	650 mμ.
Dye A (1 g./litre) ...	1·75	0·68	0·21
Dye B (1 g./litre) ...	0·11	0·23	0·32
Mixture in alcohol–water	2·07	1·75	1·30
Mixture in pyridine–water	2·08	1·37	1·17

(Figures in the above table represent optical densities in a 1-cm. cell at the wavelengths indicated)

14. The absorption spectra of three dyes—A, B, and C—in solution are indicated in the table below. Describe these colours with particular emphasis on the differences between A and C and between A and B. Would you select A or C to form the basis of a grey mixture? Give reasons for your choice.

Optical Densities of Solutions

Wavelength (mμ.)	A	B	C
400	—	—	0·30
450	—	0·05	0·75
475	0·05	0·25	1·20
500	0·35	0·70	1·75
520	0·95	1·45	2·10
540	1·75	2·00	2·30
550	2·00	2·35	2·50
560	2·30	3·10	2·70
570	2·85	3·75	2·85
580	3·00	4·00	2·90
590	4·00	3·75	2·92
600	3·00	2·35	2·85
625	1·40	0·80	2·20
650	0·50	0·25	1·30
675	0·10	—	0·60

15. Painted cards—P, Q, and R—having trichromatic coordinates in Illuminant C as shown in the table below are mounted to form sectors of a disc, each sector being adjustable in area. The disc can be spun to fuse the colours. What are the colours P, Q, and R? Disregarding lightness, is it possible by suitable adjustment of areas to match the colours L, M, and N, and roughly how would this be achieved? What is the relation between N and P?

Trichromatic Coordinates of Colours

Colour	*x*	*y*
White	0·310	0·310
P	0·500	0·310
Q	0·180	0·460
R	0·110	0·160
L	0·140	0·170
M	0·280	0·320
N	0·050	0·310

BRANCH I—TINCTORIAL TECHNOLOGY AS APPLIED TO TEXTILES

Paper C

Properties of Materials and Finished Products and Fundamental Mechanisms of the Industrial Processes

10 a.m.–1 p.m. on Friday, 10th June 1955

(*FIVE questions only to be attempted*)

1. Enumerate the chief differences, physical and chemical, between cotton cellulose and regenerated cellulose as represented by normal viscose rayon. Show that these differences may be reflected in the dyeing behaviour of the respective fibres when direct cotton dyes are applied.

2. Discuss the methods available, to dye manufacturer and to dyer, for varying the affinity of the dye in the production of vat dyeings on cellulosic textile materials.

3. Survey the chemical reducing agents employed by the dyer and the printer, and comment on the principles which may govern the choice and the conditions of use of **one** of them.

4. What aspects of dyeing behaviour are epitomised in adsorption isotherms? Give an example of one type of isotherm encountered in the investigation of dyeing systems, and show what detailed information could be obtained from it.

5. What is a mordant? Give an outline of one traditional process of mordanting, and thence show how, in modern times, analogous processes have been developed, using newly discovered chemical assistants or applying the process to modern man-made fibres.

6. What may be the functions of an ageing or steaming process in textile printing? Illustrate the discussion by examples drawn from **two** different printing styles.

7. Discuss critically the methods available to the dyer and the printer for producing or maintaining a desired hydrogen ion concentration in the bath or on the print during the different stages of the coloration process.

8. **Either** give a synopsis of an article or communication that you would like to submit to the *Journal* of the Society **or** define the scope and the contents of any book that you would like to see written upon some aspect of textile coloration.

9. Outline the chemistry of the azoic (insoluble azo) dyes. Describe the essential stages in the application of an azoic combination to (*a*) cotton and (*b*) a non-cellulosic fibre.

10. Comment briefly upon each of the following, showing the parts they play in textile dyeing processes—(*a*) hydrolysable esters, (*b*) copper compounds, and (*c*) cation-active auxiliary products.

11. "The production of a satisfactory dyeing on wool cloth depends on either initial uniform absorption of dye or subsequent dye migration, or

both." Discuss this statement with particular reference to (*a*) equalising acid dyes and (*b*) neutral-dyeing metal-complex dyes.

12. What do you consider to be the most important properties of equalising acid dyes in their application to polyamide fibres? Show how these properties influence (*a*) the application of mixtures of dyes of this class to 100% nylon fabrics and (*b*) the production of solid dyeings on wool–nylon mixtures.

Paper D

Industrial Procedures and Plant

2–5 p.m. on Friday, 10th June 1955

(*SIX questions only to be attempted. The construction and the manner of operation of any machines referred to should be illustrated with sketches where possible*)

1. Describe the dyeing of cotton fabric with a vat dye applied in (1) its reduced (leuco) form **and** (2) its pigment form. Indicate how your choice of the method to be used would depend on the structure of the fabric.

2. Discuss **one** of the following topics—

(*a*) The engraving of an intaglio printing roller from the artist's design

(*b*) The choice of blanket and backgrey as determined by style and pattern

(*c*) The function and the adjustment of box-wheels, lint doctors, and colour doctors.

3. Give a critical account of the procedure for **either** (*a*) drying dyed flat-knitted fabric to predetermined dimensions on a modern pin stenter **or** (*b*) drying dyed tubular-knitted fabric to a predetermined width and calendering continuously on **one** of the types of machine commonly used for this purpose.

4. Outline the construction of two different kinds of machine for dyeing yarn in hanks, and show how the choice of machine may be determined by (*a*) the class of dye to be applied and (*b*) the characteristics of the yarn.

5. Show how the plastic properties of a textile fibre may influence the results of (*a*) dyeing and (*b*) finishing processes. Illustrate your answer by reference to one natural fibre and one man-made fibre in each case.

6. How does hardness originate in water, and how is it defined quantitatively? When may the use of hard water assist dyeing operations, and when may it make them more difficult? Outline an industrial procedure, with the necessary plant, for reducing the equivalent hardness from 60 p.p.m. $CaCO_3$ to zero.

7. What are the essential stages in the preparation of a grey cotton cloth for printing? Give a brief account of the plant for carrying out this preparation.

8. How has the design of **either** (*a*) dye jiggers **or** (*b*) machines for dyeing loose fibres been modified in recent years, and how have the modifications increased the scope and the efficiency of the machines concerned?

9. Discuss the underlying principles of methods for colouring textile fabrics with the aid of resin binders. Compare the fastness properties of colourings obtained by the use of these methods with those of colourings obtained by conventional dyeing procedures.

10. Write a short essay on **one** of the following—

(*a*) The printing of wool slubbing (Vigoureux printing)

(*b*) Fast blacks on silk **or** viscose rayon

(*c*) Fast dyeings on half-hose.

Paper E (i)

Analytical and Testing Techniques

10 a.m.–1 p.m. on Saturday, 11th June 1955

(*FIVE questions only to be attempted*)

1. Describe briefly how you would differentiate between the types of damage caused to cotton by—(*a*) hot acid treatment, (*b*) drying-in of mineral acid, (*c*) acid oxidation, and (*d*) alkaline oxidation.

2. List the possible causes of warpway streakiness or stripiness in a pastel-dyed rayon-staple dress fabric and briefly describe how you would test for each of them.

3. Discuss the use of the polarising microscope in the analysis of fibres and the detection of faults.

4. Describe the analytical methods you would use for determining the suitability of a sample of starch for use in a printing paste.

5. State how you would determine (*a*) the amount of residual sulphur dioxide and (*b*) the presence of a fluorescent brightening agent on bleached white knitting yarn.

6. List the methods, other than solvent extraction, which are available for determining the presence and the distribution of oil and grease after scouring of wool piece goods. Describe how one of these methods may be used for rapid routine checking of the efficiency of wool hank scouring on a tape or brattice machine.

7. How would you prove the presence of active mildew on textile materials, and how would you decide whether the growth had affected the dyeing properties?

8. Describe the Society's methods for testing the fastness to washing of coloured materials, and compare their advantages and disadvantages with those of other methods known to you.

9. Describe how you would determine the following characteristics of a water supply for a dyehouse—(*a*) temporary and permanent hardness, (*b*) iron content, and (*c*) liability to set up corrosion in economisers.

10. Describe the method you would use for determining the efficiency of a continuous drying machine in terms of pounds of steam used to evaporate one pound of water from the textile material being dried.

11. How would you decide what method had been used to mothproof a wool textile? Give a method for determining the amount of a mothproofing agent on wool.

12. Explain the chief features of the system of identification of dyes first devised by A. G. Green and later modified by E. Clayton. Upon what general principles does the system depend? Pay most attention to the identification of dyes on the fibre.

Paper E (ii)

Organisation of Production and Economics

10 a.m.–1 p.m. on Saturday, 11th June 1955

(*SIX questions only to be attempted*)

1. Give an explanation of the fall in Lancashire's export trade in cotton goods since 1914.

2. Is it your opinion that the world's consumption of textiles will rise during the next decade? Give reasons for your answer in respect of (*a*) the natural fibres and (*b*) man-made fibres.

3. The Monopolies and Restrictive Practices Commission have recently condemned the practice of price maintenance in the calico printing trade. Discuss the possible consequences of this expression of opinion.

4. Piece dyeing is usually carried out on the jig, on the wince, or by continuous methods. What are the factors which influence the choice of the method employed?

5. Discuss the difficulties of processing orders in strict sequence through a dyeworks.

6*. The cost of labour as a percentage of total cost varies considerably between different sections of the textile-finishing industry. Discuss this statement.

7*. The raw material of the piece-goods finishing industry is woven or knitted grey fabric. Discuss critically a number of faults in this raw material which increase the finisher's costs of production.

8. How do the width and the weight of a fabric influence the cost of processing?

9*. In what way may the increasing use of mixtures of fibres gradually change the structure of the British textile-finishing industry?

10. Express your preference from a textile point of view for either bilateral or multilateral trade agreements between nations.

11. What kinds of corrosion are likely to be encountered in the fabric and the machinery of a dyeworks? How may the incidence of corrosion be minimised by (*a*) correct planning of the installation and (*b*) suitable working precautions?

**Note*—In Questions 6, 7, and 9 the term *finishing* is used in its widest sense to include dyeing and printing.

Report of the Diplomas Executive Subcommittee on the 1955 Associateship Examination

The 1955 Associateship Examination was held on June 9–11th in the following centres—

Technical College, Bradford
College of Technology, Manchester
Nottingham and District Technical College
Technical College, Paisley
Department of Industrial Chemistry, The University, Cairo

The thanks of the Society are due to the authorities at these institutions for the facilities made available to us.

The arrangements for running the examinations in Bradford, Manchester, Nottingham, and Paisley were left in the hands of the West Riding, Manchester, Midlands, and Scottish Section Committees of the Society respectively, and thanks are due to them and to those of their members who acted as invigilators.

Thanks are due also to the examiners, who provided not only an admirable set of papers but reports, the salient features of which are given below, which will be of much value to both students and teachers.

Twenty-six candidates entered for the examination, but five of them either failed to present themselves or failed to complete their papers, so that only twenty-one candidates completed the examination, and of this number ten took only the first part of the examination.

Paper A

Section I—General Chemistry and Properties of Dyes, etc.

In general the technique of answering the various questions in this section was not good. Several candidates had either not read or not understood the question, judging from the way in which they gave a long answer which was only partly relevant to the set question. Undefined abbreviations and symbols were used much too freely in the answers. It was somewhat surprising that Question 1 appeared to bother most of the candidates, only five of whom obtained more than half marks for this question. Neither Question 5 nor Question 6 was attempted by any candidate; this is not surprising in the latter case, as there was no candidate making a special study of lakes and

pigments, but the complete absence of any attempt from among twenty-one candidates from all over the country to answer a question concerned with the azoic dyes is most surprising.

SECTION II— GENERAL CHEMISTRY AND PROPERTIES OF HIGH POLYMERS

The general standard of the answers was satisfactory, although in a few cases there was a tendency to introduce matter of a purely textile character to the exclusion of more general and fundamental material.

Reasonable knowledge was shown of the meaning of such terms as *copolymerisation*, *cross-linking*, and *second-order transition point*, but the meanings of *number-average molecular weight* and *intrinsic viscosity* were not always clearly understood. Only one candidate, with slight success, attempted the question on methods of study of polymer structure. Such methods, which are physico-chemical in nature and basic to an understanding of the relationships between structure and properties, should be included in the reading of candidates. Most candidates gave an account of the chemistry of one of the three types of polymer cited, but the majority contented themselves with the chemical reactions involved in the production of the polymers and failed to discuss the chemical properties of the polymers themselves.

The question on modifications of the polymer chain in relation to fibre-forming properties was popular and generally well answered. Essays, though attempted by most candidates, were, except in a few cases, poorly written. Candidates must realise that an essay, even if brief, is an ordered account of the subject, and that form, as well as detail, is important. Clarity and conciseness of expression in answers generally could well be improved.

PAPER B

THEORIES OF COLOUR, ETC.

In general the papers were of higher standard than those written at the 1954 examination and showed clear evidence of preparation for the examination. There is still, however, room for considerable improvement. The greatest improvement was in the section dealing with numerical examples. In 1954 the attempts at such questions were limited and the answers very poor. Clearly, candidates had had no practical experience of the methods they described, and had never worked out a practical example. This year the position was completely reversed. Nearly all candidates attempted two out of the three questions available, and usually gave a satisfactory reply. Indeed, it appeared that the standard of the questions was lower than the abilities of the candidates warranted. The second notable point is that candidates continue to fail particularly badly on the section dealing with colour and constitution. No improvement was detectable here. Only three candidates attempted more than the single question in this group which they were forced to attempt, the majority plumping for the general question No. 10 and giving answers of rather low quality.

PAPER C

PROPERTIES OF THE MATERIALS AND FINISHED PRODUCTS AND FUNDAMENTAL MECHANISMS OF INDUSTRIAL PROCESSES IN TINCTORIAL TECHNOLOGY AS APPLIED TO TEXTILES

On the whole, the standard of the papers was better than in 1954, and an encouraging proportion of the candidates showed some capacity for seeing the connections between their practical experiences and the facts and phenomena encountered in the different parts of their studies. The chief complaint is in the lack of depth in the knowledge shown by many candidates, lack of precision in the statement of scientific ideas, and, at times, downright inability to make their meaning clear.

Several candidates had obviously left their systematic studies of textile processing behind for some time, and had neglected opportunities to revise them and bring them up to date. Where personal experience could be drawn on, it was often done very effectively. It is, however, the intention of a professional examination to test the general equipment of a would-be practitioner, and this must include his theoretical knowledge. There is otherwise no sure guarantee of his progressive attitude as his career develops.

The replies to the various questions may be analysed as follows—

Question 1 (7 replies)— There was a tendency to concentrate on the structural differences in the fibres, but the effects of these differences on dyeing behaviour were not developed as fully as they might have been. The effects of the carboxyl content were scarcely mentioned.

Question 2 (8 replies)— This question gave scope for ingenuity, and it was well answered on the whole.

Question 3 (3 replies)— The reducing agents that could be used were fully enumerated, but not all candidates were sure about the principles governing their choice for specific purposes.

Question 4 (1 reply)— The single candidate attempting this mistook rate-of-dyeing curves for isotherms.

Question 5 (9 replies)— Some definitions were too narrow. In certain cases, too much time was given to describing traditional mordanting, but there were several quite good answers.

Question 6 (3 replies)— Mostly answered fully. Candidates obviously knew what was required, but some found it difficult to display their knowledge effectively.

Question 7 (2 replies)— Here again, those who answered had the correct ideas, but found it difficult to give proper examples.

Question 8 (1 reply)— One valiant effort, in which the candidate pleaded for a book on colour, its definition, measurement, etc., with particular reference to the colouring of textiles and the properties of coloured materials.

Question 9 (9 replies)—The chemistry of the azoic dyes and their application to cellulose fibres was generally well understood. There was only one really good answer to (*b*), the application to a non-cellulosic fibre.

Question 10 (4 replies)—No candidate was equally at home with all three sections, section (*b*) being most familiar.

Question 11 (5 replies)—Two quite good answers, but several candidates failed to draw the conclusions which follow from the fundamental differences in dyeing behaviour between the two types of dye.

Question 12 (3 replies)—Not very well answered, because the candidates seemed to fight shy of drawing conclusions.

Paper D

Industrial procedures and plant employed in tinctorial technology as applied to textiles

On the whole the questions were answered clearly, with the facts set out in orderly fashion. It is necessary once again to impress on candidates the need to include in their answers all relevant facts or points (however briefly) which reveal their practical knowledge and experience of machinery and processes. Candidates could be very clearly divided into two groups—those with inadequate knowledge and those fully conversant with the subject of the paper.

Paper E

Here the candidates had a choice of paper.

(i) Analytical and testing techniques used in tinctorial technology as applied to textiles

Three candidates returned very good papers, one had a very practical approach to the questions, two others appeared rather uneven in their knowledge, and two failed.

The following comments on certain questions may be of use—

Question 1 (8 replies)—Three very good replies, but the remainder did not show clear understanding of the matter.

Question 2 (3 replies)—One very good reply and two of medium quality.

Question 3 (1 reply)—One good reply.

Question 4 (2 replies)—One very good reply and one of medium quality.

Question 5 (3 replies)—One of medium quality and two of rather low quality.

Question 6 (1 reply)—One very good reply.

Question 7 (3 replies)—The replies were of medium quality, the answers on mildew being disappointing and inadequate.

Question 8 (7 replies)—Two good, four medium-quality, and one poor reply. One paper described the Society's methods using the Marney machine instead of the Wash Wheel. The other papers were satisfactory as regards knowledge of the solutions and methods of the Society's tests. The second half of the question was not answered well by any candidate; seemingly they did not know the methods of the Swiss, German, and U.S.A. fastness testing committees. Little reference was made to the methods used in the large number of laboratories which do not possess a Wash Wheel.

Question 9 (4 replies)—One very good answer by a candidate who described the ethylenediaminetetra-acetate method, and one medium-quality reply. The other two answers were unsatisfactory. The examiner had expected that most candidates would attempt this question.

Question 10 (2 replies)—Two excellent answers. It is pleasing to see that some teaching establishment is giving attention to this type of work.

Question 12 (6 replies)—One very good, three good, one medium-quality, and one poor reply. The general impression given was that, although the tests were known, there was no clear understanding of the general principles, i.e. the approach was rule of thumb.

(ii) Organisation of production and economics in tinctorial technology as applied to textiles

Three candidates attempted this paper. They all showed complete lack of knowledge of economics as this subject is understood, and in each case avoided Questions 1, 3, and 10. In the main, however, they made a good effort to answer the other questions, but in each case there was shown a natural inclination to move away from the economic aspect to the technical or practical point of view.

Interview

All the candidates who completed their papers were interviewed by the Diplomas Executive Subcommittee either in Bradford on 7th July or in Manchester on 9th July 1955. The interviewing of the candidates has proved to be a very useful guide to the Subcommittee in its estimation of a candidate's professional knowledge and ability, particularly when the written papers have tended to show weaknesses in one or other directions.

General

After considering the examiners' reports on each candidate and having interviewed them, the Subcommittee recommended that, of the ten candidates who took Papers A and B only, nine be deemed to have satisfied the Committee and one to have failed to do so; and that, of the eleven candidates who took the full examination, eight be deemed to have satisfied the Committee, two to have satisfied the Committee in respect of Papers C, D, and E but be required to take Papers A and B again, and one candidate be deemed to have failed. (See page 544.)

The quality of the candidates who presented themselves for examination was in the main very good, and most of them showed clearly the value of the special courses in preparation for this examination that have been conducted in Bradford, Manchester, and Nottingham.

Many candidates, however, in answering general questions on fundamental science tended to avoid the generalities desired and to confine themselves to the particular aspects directly connected with their specialised work.

The inability of many candidates to express themselves either clearly, concisely, or systematically is a matter for regret, and is a reflection on the standard of general education given in our schools, for all had attained the Ordinary General Certificate of Education level in English. There is no doubt that much more attention should be devoted to the need for clarity of expression to be constantly inculcated into students in teaching establishments of all types and at all levels.

C. O. CLARK (*Convener*) J. V. SUMMERSGILL
R. L. ELLIOTT H. A. TURNER
W. PENN A. G. TYLER
C. B. STEVENS T. VICKERSTAFF

THE THIRD LONDON LECTURE

The Guilds of the City of London with special reference to The Worshipful Company of Dyers

P. E. JONES

Meeting of the London Section held at the Waldorf Hotel, Aldwych, London W.C. 2, on 4th March 1955, Mr. R. A. McFarlane in the chair

The history of the City guilds is outlined from medieval times until the present day. The craft of Dyers was mentioned in 1298, but the Dyers' Company was not incorporated until 1471, and reincorporated in 1472. Its subsequent history is traced.

The story of the guilds of the City of London is the heart of London history, the life-blood which for centuries gave the City vigour. The City owes, and has always owed, its importance to its trade and commerce, and this trade was built up, controlled, and fostered by the guilds, among them the Dyers, the senior of the junior companies. The Society of Dyers and Colourists has some objects appropriate to a City guild—to foster a craft or trade—and was founded as long ago as 1884. Since its foundation many of the ancient companies of the City have come to realise that in a modern world their institutions and activities must be moulded to the needs of a changed commercial population, a population no longer all freemen of the City and no longer members of the guilds. In recent times the Dyers' Company has given substantial support to education, in particular to Westminster School and King Edward School, Norwich, and is now closely associated with the Society for the encouragement of research in the industry. But the history of the Dyers' Company, or rather the history of dyeing in the City of London, has still to be written. As City companies were closely connected with trade, their history should be economic and technical as well as constitutional and social. Charters, grants of arms, and suchlike instruments are but milestones on a long journey, relatively unimportant in travel through seven centuries or more.

There are at present eighty livery companies in the City; the list is continually changing by additions, amalgamations, separations, and extinctions. Though combs are still used by everyone, the Combmakers company is extinct; the Tobacco Pipemakers, once extinct, was revived last year; the Master Mariners and City Solicitors have been added to the list in modern times. The companies range alphabetically from Apothecaries to Woolmen and include the Fletchers (makers of arrows), Horners, Loriners, and Basketmakers. The textile trades are represented by the guilds of Mercers, Drapers, Merchant Taylors, Weavers, Clothworkers, Haberdashers, Glovers, Broderers, Framework-knitters, and Dyers. Throughout the centuries it was the endeavour of every trade in the City to achieve its own organisation and to get recognition of that organisation for the purpose of trade control. Yet most of the guilds began as a brotherhood with few, if any, powers in relation to their trade. In the twilight of history they were fraternities, religious and social in character. The wheel has now turned full circle and many have now reverted to a social organisation, having shed their hard-won trade powers. Some guilds existed before the Norman Conquest, others took shape under Henry I, but for most it is difficult to suggest a definite date of origin—they are just found to be in existence at a particular date. Such guilds were not peculiar to London: they existed all over Europe. Except in Switzerland, the guilds on the Continent were suppressed or perished. Thus the guilds, now the livery companies, of London constitute a vigorous and almost unique survival of institutions which formerly played such an important part in the commerce of Europe.

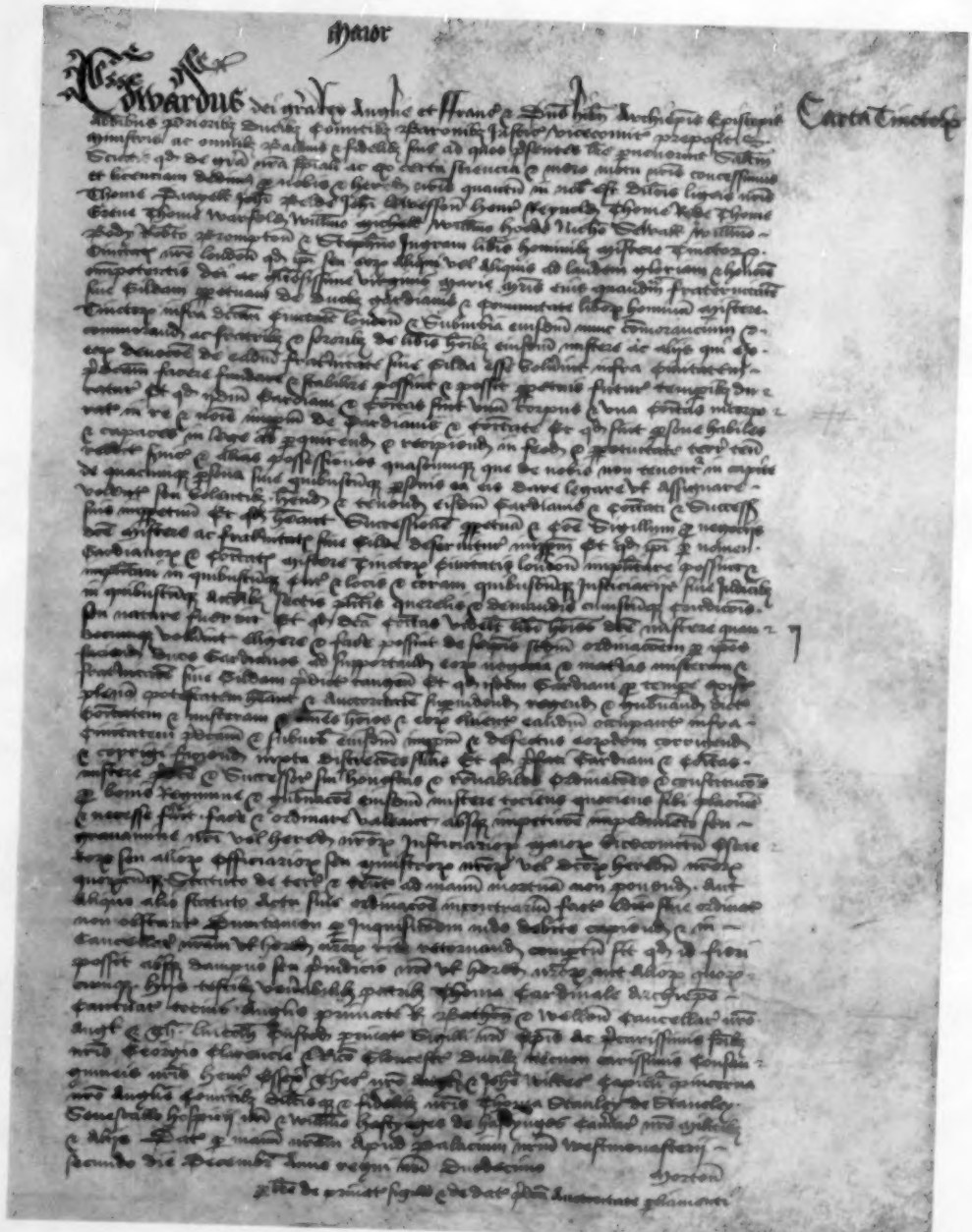

FIG. 1—Charter of the Fraternity or Guild of Dyers, 2nd December 1472

Prior

Ordinacio tinctorum

Memorandum quod nono die Julij anno regni Regis Edwardi quarti post conquestum tercio decimo Gardiani artis siue misterij Abbatis Tinctorum Cuitatis London. Coram me Johe Stokker Maiore Ciuitatis predicte coram Willmo Hariot militi ac Maiore & Aldermannis eiusdem Ciuitatis & postea videlicet xiiij^o die eiusdem mensis & Anno quarto predicto billam siue Supplicacionem cuius tenor sequitur in hec verba. ¶ To the right honorable lord the Maire and the right worshipfull Aldermen of the Cittee of London. Humbly beseeched your goode lordshipp and maisterships the Wardeyns and all the goode ffolke of the Craft of Dicers of the said Cittee. That it would please the same your lordshipp and maisterships for the worshipp of the said Cittee and for the goode rule to be had w^in the said Craft to graunte and establisshe certeyne Articles following and thereyn to be entred of Recorde in the Chambre of the yeldhall of the said Cittee before yo^r said lordshipp and maisterships hereafter. for to be vsed and executed.

ffirst that euy p^sone enfraunchised in the said Craft kepe and p^fourme all goode ordenances and rules of the same Craft auctorised by this Court. And that no p^sone of the same Craft retrete or withdrawe any ope p^sone of the ffelishipp of the same Craft vppon payne of x^li. halfe to be paied to the Chambre of the yeldhall of the said Cittee and thother halfe to the behalfe of the said Craft.

Also that euy man enfraunchised in the said Craft which entendeth to take any Apprentice shall or he bynde any suche Apprentice to hym shold hym vnto the Wardeyns of the said Craft for the tyme being to thentent that they may se and knowe that he be not lame or disfigured, for the honour of the said Cittee and worshipp of the said Craft And therevppon if yo^r be founde the Wardeyns to admitte hym and elles not. And that the said Wardeyns the contrary hereto to pay x^li. halfe to the said Chambre of the yeldhall and thother halfe to the said Wardeyns to thuse of the behalfe of the said Craft. And that euy suche Maister at habelyng of euy suche p^sone to be his Apprentice pay to his Wardeyns for the tyme being to thuse of the said Craft x^li.

Also that no man enfraunchised in the said Craft take into house of the same Craft any p^sone but suche as hath ben Apprentice a yere, and w^in a mand enfraunchised in the same Craft vppon payne of x^li to be paied halfe thereof to the Chamberlyn of the said Cittee for the tyme being to thuse of the behalfe of the same Cittee And that other halfe to the Wardeyns of the said Craft for the tyme being to thuse of the behalfe of the same Craft.

Also that euy such man in the said Craft appere afore the Wardeyns of the said Craft as often as they shalle send duely warned by the Beadell or Bailiff of the same Wardeyns to vnderstonde and knowe the goode rules of the same Craft vppon payne of forfaiture of x^li to be paied vnto the same Craft.

Also that euy p^sone of the said Craft makyng defaute or not comyng to the place and at houre limited to the Wardeyns of the said Craft when they shalle be thereto duely warned in mand aforesaid for to worke vppon my lorde the Maire for the tyme being. for the deth or byrthyng of any p^sone of the said Craft or for mariage daies or ope cause reasonable pay x^d. halfe to the Chambre of the said Cittee and that other halfe to the said Wardeyns for the tyme being to the vse of the behalfe of the said Craft as often as such case shall happen reasonable cause of Excuse onely except

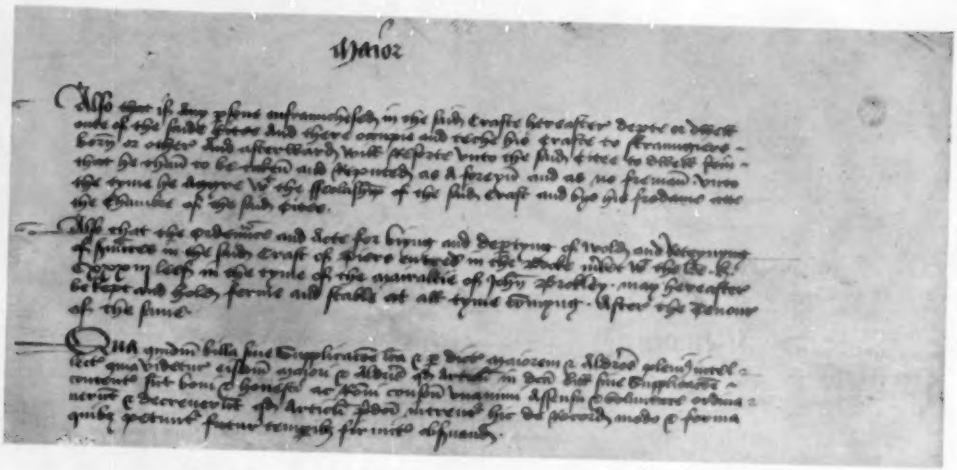

Fig. 2—Ordinances for the Good Rule of the Craft of Dyers, 9th July 1482

The Dyers of London were first incorporated on 16th February 1471 by Henry VI (Patent Roll) and reincorporated by Edward IV on 2nd December 1472 (Charter Roll—Fig. 1). The first charter was obtained during the short restoration of Henry VI, who died at the Tower in May 1471. The question arises whether the Dyers favoured Henry or whether they took advantage of his need for money. However that may be, it cost them dearly, for on the restoration of Edward they had to buy their incorporation again and to be satisfied with lesser privileges. The charters constituted the Dyers a fraternity or perpetual guild to the glory of God and the Virgin Mary, consisting of two wardens and a commonalty of free brothers and sisters, with a common seal and power to make reasonable and honest ordinances for the government of the mistery. The first charter gave them power to search merchandise and works pertaining to their trade and to seize false goods within twenty miles of the City; the second merely gave their wardens power to survey, rule, and govern the mistery and all men and their servants using the trade in the City and suburbs. Later charters settled the area of jurisdiction as ten miles around the City. The twelve freemen named in the first charter were Thomas Danyell, Henry Reynold, Thomas Rede, Thomas Grene, Thomas Warfeld, William Michell, William Hoode, Nicholas Sewall, William Elmet, John Beale, John Haydon, and John Lowes; the last four were replaced in the second charter by John Belde, John Lewesson, William Body, Robert Brompton, and Stephen Ingram, making thirteen. The ordinances made under the charter were submitted in 1482 for approval to the "right honourable lord the Mayor and the right worshipful sovereigns the Aldermen of the City of London" by petition of all the good folks enfranchised in the craft of dyers (Fig. 2).

But the mistery of Dyers was recognised long before 1471. When the City Council was elected by the guilds in 1381 the mistery of Dyers appointed two members, Nicholas Maynard and Walter Anne. Brokers of woad were chosen by the mistery of Dyers in 1343, and the craft of Dyers is mentioned in connection with the control of the fulling of cloth in 1298. Research would doubtlessly produce earlier references, and might establish whether the dyers broke off from the weavers or perhaps from the fullers. In Florence the dyers were first a religious brotherhood within the guild of weavers of that city and were subject to the powerful cloth merchants. They broke away in 1371 and were acknowledged as a separate craft in 1377. In Venice it was not till 1429 that the dyers were recognised as a separate craft, and in Germany, although a distinct guild in the tenth century, they became a subsidiary of the weavers till the sixteenth century. Thus in London it appears that the Dyers achieved recognition as a separate mistery, although unincorporated, before their colleagues on the Continent. In England illuminated manuscripts show that the coarse dress of the early Middle Ages had given way to finery and colour, and it was probably this change that enabled the Dyers to enhance their status.

There were two sides to a guild or company—the fraternity or religious and social side and the craft or trade side. The fraternity was often associated with a patron saint and generally attached to a particular church. The Grocers are the Fraternity of St. Anthony, and the Merchant Taylors the Fraternity of St. John the Baptist; the Dyers might have been the Fraternity of the Blessed Virgin Mary. Most companies still attend church on some day of moment in their history. It was a duty to attend the funeral of a member of the guild. Gifts to a company were often coupled with prayers for the soul of the donor, involving attendance at church or a gift to a priest. The social side included care of the sick and infirm and the poor of the company and their dependants and gifts of a charitable nature over a wide field. The Dyers' almshouses in City Road and Spitalfields were the result of bequests, and the Company also administers trusts for its poor and for the poor of the parishes of Allhallows Thames Street and St. Martin Vintry.

From medieval times the dyeing industry in London had been located in this area between Cannon Street and the Thames. In 1373 Sir Edmund Cheyne Kt. owned a dyehouse in the parish of St. Swithin. A tenement called "le Dyhouse" in Thames Street, parish of St. Peter, was let to Thomas Bottyng, dyer, in 1457 by John Ludford, dyer, and adjoined the house of Thomas Wayfeld, dyer: obviously a colony of dyers. Attached to "le Dyhouse" was a wharf, so necessary for the industry in those days. The medieval industry is well illustrated by two cases. An action was brought in 1357 against Thomas de St. Edmunds for carrying lengths of wet cloth in the process of dyeing through a passage in the house whereby water dripped and penetrated into the plaintiff's rooms. No doubt he was taking his cloth from the dye to the dip. Thomas pleaded that the property had been used as a dyeworks since 1303 and had always been let subject to the right of passage. Another action of nuisance by dyeing was settled in 1417 by arbitrators. William Neubolt, dyer, was the tenant of Richard Chipenham, tailor, who complained of a pewe (puteus) or pit before the door of the house into which the dyer put hot cloth after it had been dyed. The arbitrators, a fuller and a tailor, awarded that an oven within the tenement should be converted into a pit for hot dyed cloth and that the tenant ought to have the advantage of a gutter leading the water from the dyehouse to the Thames.

It is recognised that London's pre-eminence in commerce is due to the Thames. The river was no less necessary to the dyeing industry, particularly when, in medieval times, London's pumped water supply was non-existent or later depended on standards in the streets. A humble petition of the Dyers to the Lord Mayor and Aldermen after the Great Fire of London stated that they had

from time immemorial enjoyed stairs and "put galleys" on the banks of the river for the exercise of their trade and protested against any proposal for an open wharf or embankment from the Tower to the Temple. Although the Oxford English Dictionary defines a "put galley" as a lever for raising water, it is possible that it might have been a place from where cloths could be put into the water, similar to the put or pit outside the house complained of in 1417. Both put galleys and pot galleries were presented as encroachments on the Thames and were often owned by dyers, so they may be identical, although the dictionary does not associate them. Part of the fascination of ancient documents is to be found in their use of obsolete words and phrases, and documents might be recommended as a change from crosswords, clues to meaning being just as intriguing.

When did the medieval guilds come to be called *livery companies?* What was the significance of this change? Dress consciousness not only boosted the trade and raised the status of the dyers, but also divided the guilds into classes; it created an aristocracy in each guild and profoundly affected the government of the City. All this happened because the wealthy citizens in the fourteenth century began to array themselves in a distinctive dress on solemn or festive occasions. Originally the wearing of a distinctive dress by the members of the guilds was purely a matter of domestic concern; all the grocers could afford to wear a livery in 1345, probably none of the dyers. This guild clothing was termed a "livery" probably in imitation of the liveries adopted by the barons and lords of the realm. Originally livery signified an allowance in provisions and clothing, and gradually came to be a badge of service or protection. In London it consisted of a gown, often of two colours, and a hood, which denoted the guild of the wearer. Some guilds, such as the Mercers, also adopted a cap or hat. Apparel has always exercised a great influence in dividing persons and classes from a social standpoint, and in London a distinction grew up between guilds whose freemen could afford a livery and those whose trade would not permit such luxury. The fortunate became livery companies, the others remained just City guilds. The Horners, surely a very ancient trade, did not aspire to a livery till 1846, long after horn vessels had ceased to be used, and the Basketmakers obtained a livery in 1825. By 1502, 47 of the 75 guilds had adopted a livery. Within some of the companies some freemen could afford and would be admitted to the livery, others could not and must await the honour. Henceforth the membership of a livery company consisted of liverymen and freemen. More recent grants of livery have limited the number of liverymen to be admitted, but the Dyers are one of the ancient livery companies with an unlimited livery.

The recognition of a guild by the City enables its freemen to be admitted to the freedom of the City as "Citizen and Dyer", "Citizen and Draper", etc. The adoption of a livery not only enabled the liverymen of a company to play a full part in City ceremonial, but from 1475 to the present day only those freemen who were liverymen had the right to elect the Lord Mayor and Sheriffs. Also, until 1832 the liverymen alone elected the Members of Parliament for the City. The livery vote in parliamentary elections was not finally abolished until 1948. A City company consisted normally of apprentices, journeymen, freemen, liverymen, assistants, wardens, and master. It was usual for some fee to be payable at each step. A prime warden replaces the master in the Basketmakers, Blacksmiths, Dyers, Fishmongers, Goldsmiths, and Shipwrights, and the master of the Weavers is the Upper Bailiff. A company is governed by a Court of Assistants. The powers of the Dyers' court were defined by their charter as to determine, execute, and despatch all matters concerning the Company, and to consult and confer together about the statutes and ordinances of the Company, and the said good government thereof, according to their discretion. The court enforces the powers conferred by charters and ordinances, which in past centuries enabled it to control the trade and all persons exercising the trade not only in the City but also in the suburbs.

Perhaps the most interesting part of a company's history is the endeavours it made to control the trade. Each company sought a monopoly. As regards operatives they were the medieval trade unions, but they were concerned also with supplies, demand, workmanship, and the good name of the trade. Unlike the modern unions they were not concerned with labour only, but with raw materials, craftsmanship, and the price and quality of the finished article. The wardens had power to inspect all workshops and all wares offered for sale; they could confiscate defective goods and cause defaulters to be imprisoned. Price rates were suggested by the company, to the Privy Council or to the Court of Aldermen, and enforced by the search and inspection of the company. A few examples of the exercise of trade control by the Dyers will illustrate the system.

Woad was originally the universal dye, the colour depending on the strength of the bath and the number of dips. In the Middle Ages it was imported into England from France, Flanders, and Germany. Shipments are noted in 1332 from Amiens and Corbie, and a cargo of 37 tons arrived in London from Calais in 1366. A large quantity of woad belonging to French merchants in London was arrested in 1370 as a result of war between France and England. On the Continent woad was in the hands of the weavers, an indication of their power over the Continental dyers. In England the mistery of Dyers petitioned in 1433 against the cornering of woad by certain men to their own profit. An order was made that no-one should buy woad except the wardens of the Dyers and that they should make a fair distribution among all members both poor and rich. Again in 1501 it was ordered that only the wardens of the craft of Dyers should buy woad from merchants, to the end that every man of the craft, poor or rich,

might have his portion. Thus for a long period woad was a monopoly of the guild. The growing of woad in England was not encouraged, and a proclamation of 1586 was issued against breaking up of ground for planting woad. By that time, however, indigo was ousting woad, and from the fifteenth century onwards brazilwood, madder, saffron, and bark dyes from the Indies in the east and America in the west were growing competitors. The aforementioned petition of 1501 also complained that "Lytmose and Orchell" (orchil), dyes obtained from lichens, were engrossed by certain persons and sold at their pleasure to their own profit, so that poor men had been greatly hurt "as God knoweth". So through the centuries the guild of Dyers made efforts to ensure supplies of raw materials and also fair distribution among the trade.

Rather naturally the Dyers looked with some doubt on new processes. As late as 1613 it was a presumption to pretend that colours dyed with "cutchenele" were equal to "grayne" colours. A committee reported in 1554 after search and trial that it was impossible to make a good and profitable colour of any white cloth with galls only except the same cloth be first woaded. In 1501 the guild had complained by petition that certain persons were dyeing caps and cloth with "copras" and galls to the great deceit of the purchasers and the great noise, slander, and rebuke of the craft. It was wont, they said, that caps should be first woaded and then blacked. It was ordered that no caps in London or Southwark should be dyed "half maddered and half grayned". The use of logwood was forbidden by constant orders from about 1600, and the City and the wardens of the Dyers supervised its destruction by burning. Nevertheless it has continued to be used, and has been found suitable for dyeing synthetic fibres.[1]

Nearly all City companies complained from time to time of bad workmanship and unfair competition from the inhabitants of Southwark, across the river. In 1523 the dyers alleged that their living was being taken away by the foreign dyers in Southwark, whose dyeing appeared right pleasant, orient, beautiful, and sure, but faded in a few days of wearing. Also they dyed in one work more than could be done well and substantially. In spite of complaints against and opposition to all dyers who were not free of the Company, the guild did not oppose the admission of Continental dyers from whom new secrets of the craft could be learned. In 1556 John Drake and Robert Popell, the wardens of the Fellowship of Dyers, petitioned that Henry Van Tynen and Leonard Busshe, aliens, excellent, expert, and cunning in the dyeing of woollen cloths, should be admitted to work in London as servants of some freemen of the Company, it being a monstrous loss to the trade if an exception was not made in their case. Again in 1611 the Company agreed that Guilliam Van Dam, a Dutchman, should be allowed to continue his trade in London as a thread dyer, as none of the Company had skill in that trade. He was allowed to continue on his promise to instruct Englishmen in his art and not to sell his thread by retail.

To ensure responsibility for work, good or bad, it was the custom in many London trades to require an identification mark. Everyone is familiar with goldsmiths' and silversmiths' marks, but it applied much more widely, to armourers, gunsmiths, coopers, and dyers for example. In 1501 the Fellowship of Dyers complained that of late, when search was made of cloths and kerseys to see that good colours were made, they found London dyeing whereof the colours were neither good, sure, nor able, but were untruly wrought. And forasmuch as the mark of the dyer who dyed them was not made upon them, the wardens could not know who dyed them. It was thereupon ordered that no cloth leave the house of a dyer unmarked with the mark of the dyer, that the maker of evil colours might be known. In the next year the Dyers agreed with the Drapers, Taylors, and Haberdashers that they would purchase a seal for marking all cloth that was London-dyed, that it might "be knowen for the best, surest and most profitable for the king's liege people of any dying within this Realm". It was also agreed between the guilds that the Dyers could search all cloth for false and untrue dyeing, even that in the hands of merchants not free of their company. The result of infringements of such marks is shown by the case of Mathew Waldram, who was presented by the Company for persisting in dyeing "bayes and kersies" in false colours bearing the mark of true dyed cloth to the deceit of the purchaser and the discredit of London commodities. He was fined and also imprisoned in Newgate for ten days.

The test of good colours was assured by samples which the Dyers agreed to supply in 1502 to the Drapers, Taylors, and Haberdashers. When these samples in every colour were delivered in mid-Lent 1503, the other guilds agreed to give all their work to freemen of the Dyers' Company, who in turn contracted to "work, dye and greyn" all wool, cloth, hats, caps, and yarn sent to them by the other guilds as well as, or better than, had formerly been done by anyone, and contracted also that their servants would always be ready to fetch, receive, and safely keep such goods. The Dyers agreed further not to dye for foreigners, except remnants under 10 yd. and liveries for particular households. It was also provided that the samples should be renewed as often as necessary and that disputes should not be taken to law but settled by the wardens. Here may be seen in embryo a court of arbitration and a British Standards Institution.

Most disputes seem to have concerned payment. It was the general rule that payment was made on the weight of the material when dyed and should not be due until the material was dry. As early as 1362 it was provided that dyeing should be done at the rate of 10 lb. for 8 lb., and that, although the cloth might be moved by the owner when

wet, it should be paid for so soon as it was dry, according to the weight of the wool when dry. Naturally there were many orders against undue increase of weight in dyeing. In 1611 an Act of Common Council was passed against the increase in weight of silk dyed black, and in 1614 it was suggested that a sworn viewer of silk should be appointed to detect mixtures used in dyeing to increase weight, that no dyed silk should be imported, and further that only persons apprenticed to the trade should be permitted to dye silk. Another Act of Common Council, in 1550, had attempted to ensure prompt payment of the Dyers, who, to the great annoyance of the Drapers, Taylors, and Clothworkers, resolved to publish in their Company Hall and in their dyehouses the names and the companies of all persons who had not paid by the end of each quarter their accounts for dyeing. In 1561 a compromise was concluded between the companies. The wardens of the Dyers were to inform the wardens of the other companies of the names of defaulters and the names would not be posted. It was also agreed, to avoid actions at law, that compensation to be paid for cloth marred during dyeing should be settled by the wardens of the companies concerned.

Most companies in the City have attempted at times to restrict trade, often to maintain prices or to restrict excessive competition. In 1561 the Dyers sought to forbid dyeing for twenty days after every quarter day. An ordinance of 1523 provided that no warden should dye in any one week above 14 whole cloths, accounting 4 "karseies" for a cloth, 3 broad dozen or 6 narrow dozen for a cloth, 8 dozen caps or hats for a cloth, or 7 stones of wool at 8 lb. the stone for a cloth, and no other household dyers should dye more than 12 cloths in a week, excess work being assigned by the wardens to other dyers. Fair shares of the work! Within a few months this order was revoked by the Court of Aldermen, probably as in restraint of trade. Regulations of a similar character were made in relation to the dyeing of leather. Upon a petition of the Leathersellers and Pouchmakers in 1372 it was ordered that no dyer should dye any leather for any person except the owner of the leather, nor use the dye set apart for one person for the leather of another, nor sell brasil dye for more profit than 2d. a lb., nor receive for his labour for 1 lb. of brasil dye more than 10d.

Such are a few of the many references in the City's records to the trade of the dyer, illustrating the control and regulation exercised by the guild and City authorities. Among other sources of information for a history, the inventories of the estates of dyers, or persons connected with the dyeing industry, might yield valuable details of trade, such for example as the inventory of the warehouse and customers of John Ramsey, dyers' warehouseman or drysalter, of 1669. His stock of dyes was valued at £4,485 15s. 3d., a large sum at that period Some items from the stock are worthy of transcription as examples—

Two remnants of Lahore Indigoe containing nett two hundred sixty five pounds and a halfe valued at 5s. 9d. per pound comeing to	£76 6s. 9d.
Barbadoes Indigo containing nett 32 lbs. at 4s. 7d. per lb.	£7 6s. 8d.
Quantities of round, fine flat, coarse flat and dust indigo valued at	£80 17s. 0d.
Eight barrels of Lahore Indigo in the East India House worth	£694 0s. 3d.
Cropt Mathers [Maythe, camomile], 8 bales	£262 14s. 10d.
Other sorts of Mathers are listed as Gusly, Mull, Gamene and No. 0	
Alum, English at 25s. 6d. per cwt. and Rhenish at 21s., valued at ...	£207 13s. 0d.
Shomack [sumac for dyeing leather] at 6s. 6d. and 10s. per cwt., valued at...	£221 6s. 7d.
Brazeel, young whole at 58s. per cwt.; Bahua, at 32s.; ground at 52s.; the whole valued at	£622 2s. 4d.
Redwood, whole at 66s. per cwt., ground 57s.	
Logwood, whole, 46s. 6d. per cwt., ground 40s., cory 22s.	
Sweetwood, whole 10s. per cwt., ground 6s.	
Campeach [campeachy], 22s. per cwt.	
Sapam [sapan], 18s. per cwt.	
Fustic(k), young 16s. per cwt., ground 7s., whole 5s.	
Gauls [gall], Smyrna 44s. per cwt., Alleppo 48s., ground 30s., garble of, 20s.	
Barberryes [berberis], 14s. per cwt.	
Turmeric(k), ground 50s. per cwt.	
Argall [cream of tartar], red 26s. per cwt., white 28s., Rhenish 48s., Galesia 30s., Portugal 14s.	
Copperas, 5s. 6d. per cwt.	
Litmus, 28s. per firkin.	
Silvester [inferior kind of cochineal, supposed to be the seed of a plant], 4s. per lb.	
Arnatto [anatta], 1s. 10d. per lb.	
Cochaneale, 23s. per lb.	
Verdigrease, 15d. per lb.	

John Ramsey also held a lease from the Crown of an alum works or mine at Greenhurst in Salmsbury (Salesbury) in Lancashire. The long list of debts owing to him, amounting to £30,500, shows that he did business with dyers not only in London and Southwark but also in almost every county of England and in Scotland, Ireland, Amsterdam, and Rotterdam. He had customers in Berwick, Newcastle, Leeds, Dublin, and Norwich. Among the persons to whom he owed money may be noted Jonathan Welsh, brazel grinder. From such information, his business, just after the Great Fire of London, can be reconstructed. Other similar inventories survive. The house of Philip Hudson, dyer, who died in 1667, contained coppers, brass furnaces, wooden horses, tenters, hogsheads of copperas, refined salgem (sodium chloride), potash, alum, argol, starch, cochineal, and silvester.

Why were these inventories compiled? The persons concerned were freemen of the City of London, and the custom of London required that the dependants of a freeman, in particular the orphans, should be protected. The livery companies, too, have a custom of brotherhood which is enshrined in their history and ordinances. The

Dyers' first ordinance of 1482 (Fig. 2) was "that no person of the same craft revile or rebuke any other person of the same craft". The fourth ordinance required journeymen to attend before the wardens of the Company to be instructed that they might understand and know the good rules of the same craft. The orders relating to apprentices not only ensured able craftsmen by years of instruction and experience, but required that before being bound the apprentice should be shown to the wardens to the intent that they could see "that he be not lame or disfigured for the honour of the said City and worship of the said craft". So through the centuries the endeavour of the guilds and livery companies has been, not their own ease and profit, not a monopoly or union solely for the benefit of themselves, but a brotherhood or association for the honour and good name of the craft, the honour of the City of London, and the good service of the citizens. In attempting to carry out these objects they have had experience of most modern-sounding techniques—strikes and lock-outs, closed shops and restrictive practices, price rings, monopolies and cornerings, foreign labour and dilution—they have all been present, quite recognisable, though under other names.

Today the livery companies rarely receive the praise to which most of them are entitled. The halls of the companies, for example, are usually identified with feasting, but this is largely a fallacy. Even so, feasting often oils the wheels of trade and commerce. The everyday work of the Clerk and his staff is entirely overlooked, simply because it is normal and provides no headlines. It is true that the companies are proud of their halls and delight to entertain and to honour the eminent of the land. Such halls as survived the wars are now used much more frequently, for they are loaned to less fortunate companies and let to City institutions and organisations of various kinds for dinners and lunches, thus providing an appropriate background for civic affairs. In its everyday business the hall, or company office, is a combination of an estate office, a charity organisation, an educational trust, and a clerk's department, sometimes incorporating a trade association. Some companies still have considerable trade functions, notably the Brewers, Goldsmiths, Fishmongers, and Gunmakers. All the greater and many of the minor companies are trustees of charitable bequests, which, under the general superintendence of the Charity Commissioners, are devoted to the support of schools in various parts of the country, to educational grants, to scholarships, to almshouses, to the relief of necessitous freemen and their families, and in modern times particularly to technical education, especially in the craft from which the company takes its name or in kindred trades. The Horners, for example, are identified with the plastics industry, the Coachmakers with car bodies, and the Carmen with the motor trade. Acting either alone or in combination, certain companies have founded technical or training schools, such as the National Leathersellers' College. In combination the Livery Companies have contributed £1,500,000 to the City and Guilds of London Institute for technical education. Other companies hold exhibitions of craftsmanship, issue certificates of competency, or award prizes and medals. Most disburse far more than their trust funds on such objects.

The Master and Wardens of the companies still accompany the Lord Mayor and Sheriffs to the Royal Courts of Justice on Lord Mayor's Day. The companies used to provide the tableaux for the land show and the barges for the water procession. Indeed, they contributed to the pageantry of London in many ways. In 1737 and 1738 the Dyers failed to attend the procession on Lord Mayor's Day, and the Wardens were called before the Court of Aldermen to explain their absence. They said that the Company had no hall, barge, or ornaments and were very much in debt. The Court did not accept their excuse, and ordered that in future they should attend and that the Stewards of the Company should provide entertainment for their members on Lord Mayor's Day. In past centuries the companies lined the streets on ceremonial occasions, and a strict order of precedence was settled and enforced by the Court of Aldermen. First were the Great Twelve, who for many centuries provided all the Aldermen and Lord Mayors of the City. In October 1514 it was ordered that the Dyers should stand in twelfth place next after the Vintners. Thereupon a grudge arose between the Dyers and the Shearmen "by reason of divers unfitting words surmised to be said by certain persons of either of the said fellowships in reproach of the other". An order was made that the wardens of both fellowships should give the names of those who spoke the words to the Lord Mayor. Whether as a result or not, the precedence of the companies was settled in January 1515 as Shearmen twelfth, Dyers thirteenth. The Shearmen, with the Fullers, became the Clothworkers, and the Dyers have remained thirteenth.

Today the membership of the companies is voluntary, trade in the City is no longer confined to freemen, a privilege finally abolished just a century ago, and liverymen and freemen of the City are now a representative cross-section of the community and in no way confined to London. New occupations still continue to seek recognition of their guilds by the City. The companies endeavour to foster comradeship among their members, and citizenship in the great City to which they have sworn obedience and, in their freedom oath, promised to save harmless, and above all to be of service to the community in their own sphere. It is still important that trade and technical organisations—City guilds, societies such as this Society, federations, and trades unions—should continue to accept public responsibilities and public duties as part of their objects. The public good is the supreme and noblest object.

RECORDS OFFICE
CORPORATION OF LONDON
55–61 MOORGATE
LONDON E.C.2

(*MS. received 13th April 1955*)

The Dyeing of Terylene Polyester Fibre with Disperse Dyes above 100°C.

A. S. FERN

Meeting of the Midlands Section held at the King's Head Hotel, Loughborough, on 17th November 1954, Mr. S. R. Meadows in the chair

The stability, dyeing, and fastness properties of a range of disperse dyes have been studied on Terylene polyester fibre at temperatures up to 120°C. under laboratory conditions. The potential advantages and disadvantages of dyeing under such conditions on the large scale are pointed out.

Introduction

Some textile processes, e.g. the kier boiling of cotton and the "cottage" steaming of printed fabrics, have been operated at temperatures above 100°C. for many years. The first dyeing process under such conditions seems to be that developed by the Bachmann–Uxbridge Worsted Corporation [1, 2], who in 1938 successfully processed wool pieces in steam at superatmospheric pressure under laboratory conditions, further large-scale work being carried out in conjunction with the Calco Chemical Division of the American Cyanamid Co. from 1941 onwards. Interest in high-temperature dyeing is usually thought to have quickened with the introduction of hydrophobic synthetic fibres, but the first of these which had any textile importance, from polyvinyl chloride or chlorinated polyvinyl chloride, softened excessively even at 100°C.[3]. The polyamide fibres (6,6-nylon and 6-nylon) could be successfully dyed at 100°C. or below, and although slight advantages have recently been reported [4–7] for dyeing these fibres above 100°C., there was initially no incentive to resort to a dyeing process which was, at the time, unconventional. It was not until the highly crystalline and oriented fibres from polyethylene terephthalate (Terylene, Dacron) and polyacrylonitrile (Orlon) appeared that high-temperature dyeing became of widespread practical value or, indeed, necessary.

Although Terylene has a high capacity for disperse dyes [8], the rate of diffusion of the Dispersol (ICI) and Duranol (ICI) dyes into the fibre is too slow at temperatures up to 100°C. for many of them to be of much value. Three solutions to this difficulty have been proposed—

1. MANUFACTURE OF DISPERSE DYES OF SMALLER MOLECULAR VOLUME

Such dyes would diffuse into the fibre more rapidly, but would also tend to migrate from the fibre more readily under the influence of heat or wet treatments, and are of little practical value. This technique is, however, used in the azoic dyeing of Terylene, where relatively small molecules are applied to the fibre from aqueous dispersion, and later combined by a diazotisation and coupling process [9].

2. USE OF A "CARRIER"

A "carrier" is an organic dyebath assistant which accelerates the rate of dyeing of disperse dyes on Terylene at 100°C., so that heavy dyeings are obtainable in a practical dyeing time. There are many disadvantages associated with "carrier" dyeing, among which are—

(*a*) Cost, since the carrier is thrown away after dyeing
(*b*) Potential unlevelness, particularly in the dyeing of piece-goods
(*c*) Relatively complex and lengthy dyeing process
(*d*) Difficulty of complete removal of carrier, and effect of the residue on light fastness
(*e*) Smell and/or toxicity
(*f*) Effect on other fibres in Terylene mixtures.

It is not suggested that any one carrier necessarily possesses all these defects, but it is realistic to regard carrier dyeing only as a temporary expedient which enables Terylene to be dyed in many types of existing equipment capable of achieving a maximum temperature of 100°C.

3. RAISING THE TEMPERATURE

The rate of diffusion of disperse dyes into Terylene increases rapidly as the temperature of dyeing is raised [5, 6, 10]. Qualitative experience indicates that many disperse dyes give dyeings at 110–115°C. which are similar in depth to those produced with a carrier (e.g. Tumescal D (ICI)) at 100°C. High-temperature dyeing, therefore, offers a convenient alternative to carrier dyeing, and may, if a sufficiently high temperature is possible, produce results which cannot be achieved with any known carrier. It will be obvious that many of the disadvantages of carrier dyeing listed above are eliminated if dyeing is carried out at 120–130°C.

At this stage it is desirable to consider what high-temperature dyeing can, and what it cannot, be expected to do.

At temperatures much above 100°C. the Terylene fibre must expand, and the thermal agitation of the polymer chains will increase. This implies that the statistical chance of a "pore" existing through which dye molecules can pass will also increase [11]. Thus not only should dyes diffuse more rapidly into the fibre, but they may be able to penetrate into regions of the fibre which are inaccessible at 100°C.; this phenomenon could result in increased building-up and fastness properties. Another probable effect of high temperatures is an increased rate of solution of the disperse dye in the aqueous phase. Assuming that

dyeing takes place from a saturated solution of the dye in water [12], a mechanism widely accepted for the dyeing of acetate rayon with disperse dyes, this will also account, in part, for a more rapid rate of dyeing. The third implication of high-temperature dyeing is that the exhaustion at *equilibrium* should progressively diminish as the temperature is increased, although particle size, shape, and solution characteristics may mask this effect to some extent [13]. Lastly, in common with other dyeing systems, disperse dyes should tend to migrate more rapidly at higher temperatures, thereby improving the levelling properties [1, 6, 14].

The object of the present investigation was to study the dyeing properties of a range of disperse dyes, under controlled laboratory conditions, and hence to see how far these expectations could be justified on a quantitative basis. A limited amount of work of this type on Dacron (DuP) polyester fibre has already been done [15]. High-temperature dyeing processes will become increasingly popular in the next few years, and it is important also to discover how any improvements they may offer in dyeing behaviour can be best exploited, and whether any potential disadvantages exist for which the dyer might be otherwise unprepared. It has recently been suggested [16] that a relationship between dye constitution and dyeing behaviour of polyester fibres be sought, to stimulate research into the production of new dyes. No attempt has been made to do this with the present data, as it is considered that the small number of dyes examined, and the lack of a simple or regular constitutional difference between them, would make any relationship of this type an extremely tenuous one.

Experimental

1. MATERIALS

Terylene Polyester Fibre

(*a*) 70 denier/34 filament micro-dull ordinary-tenacity filament yarn

(*b*) 60–65 denier/33 filament bright ordinary-tenacity filament yarn (levelling tests only)

(*c*) 2/28s hosiery-twist staple fibre yarn (for qualitative tests)

All fibre was scoured before use in a solution of Lissapol C Paste (ICI) (1 part) and ammonia (sp. gr. 0·880) (2 parts) in water (1000 parts) at 75°C. for 15 min. and rinsed thoroughly in cold water.

Dyes

Commercially available powder brands.

Chemicals

Analytical reagent quality.

2. FIBRE STABILITY

Samples of micro-dull yarn were treated (at 20 : 1 liquor : goods ratio) at (*a*) pH 7·0 (distilled water), (*b*) pH 2·8 (lactic acid), and (*c*) pH 11·2 (sodium carbonate) with saturated steam at pressures of 10, 30, 50, and 70 lb./sq.in. for 45 min. The tensile strength and the percentage extension at break were determined on the rinsed and conditioned yarns by means of a Cambridge fibre extensometer. This hitherto unpublished work was done by Mr. A. Scott Wild in 1951.

3. DYE STABILITY

Aqueous dispersions of the disperse dye powders (1·66 g. of the 300 Powder Fine brand or an equivalent amount of other strengths and 1 g. of Lissapol C Paste per litre) were heated in a closed stainless-steel pressure vessel at 135°C. for 2 hr. The optical absorption curves (250–600 mμ.) of the unheated and of the heated dye in methanol were obtained on a Carey spectrophotometer and compared for height and shape at equal concentrations.

4. DYEING EXPERIMENTS

With one or two early exceptions, rate of dyeing, build-up, and pH sensitivity tests were carried out by immersing Terylene micro-dull filament fibre (2 g.) in an aqueous dispersion of dye (80 ml., containing 0·08 g. Lissapol C Paste) under the appropriate conditions of concentration, time, temperature, pH, etc. in a stainless-steel "bomb" which could be rotated continuously in an atmosphere of saturated steam at the required temperature throughout the dyeing experiment. The dyebath was chilled rapidly, and the fibre removed and rinsed alternately in cold distilled water and cold acetone to remove dye adhering to the surface. The dye was extracted from a known weight of fibre by successive treatments in boiling (mono)chlorobenzene, and estimated optically in the normal manner.

The initial dyebath pH was adjusted with sulphuric acid or sodium carbonate. This procedure was considered to correspond more closely to practical conditions than the use of buffer solutions.

Levelling tests were made by treating equal weights of dyed (medium depth) and undyed bright filament yarn in a bath containing only Lissapol C Paste (2 parts per 1000 parts of water) at 120°C. for 1 hr. The proportion of dye migrating under these conditions was measured optically and expressed as—

$$\text{Migration (\%)} = \frac{\text{Wt. of dye on originally undyed yarn}}{\text{Wt. of dye on originally dyed yarn}} \times 100$$

For comparison, similar tests were made on dyeings in presence of 8% (on fibre weight) of Tumescal D (ICI) as dyeing assistant, levelling also being carried out in presence of 8% Tumescal D, and on pale or medium dyeings (depending on the building-up properties of the dye) at 100°C. in absence of an assistant.

The compatibility of dye mixtures was assessed by applying the same ratio of the components to staple yarn in increasing amounts at 120°C. for 30 min. (liquor to goods ratio 40 : 1).

Fastness tests were made on staple yarn in medium depths. Dyeings made with the aid of a carrier were given a "reduction-clearing"

treatment after dyeing; i.e. they were treated in a solution of sodium hydroxide (2 parts), sodium hydrosulphite (2 parts), and Lissolamine A 50% Paste (ICI) (2 parts) in water (1000 parts) at 70°C. for 15 min., and then rinsed in a liquor containing Lissapol C (1 part) in water (1000 parts) at 70°C. for 15 min. Dyeings made at 120°C. were merely rinsed in warm water after dyeing.

Dyebath exhaustions were calculated from the amounts of dye absorbed by the fibre in relation to that originally added to the bath. This method overcame the difficulty of measuring accurately the final dyebath volume.

Results and Discussion

1. FIBRE STABILITY

Fig. 1 shows the effect of saturated steam at 10–70 lb./sq.in. (115–160°C.) on the average toughness factor of Terylene yarn immersed for 45 min. at various initial pH values.

Average toughness factor =

$\frac{1}{2}$ × Tensile strength (g./denier) × Extension (%) at break

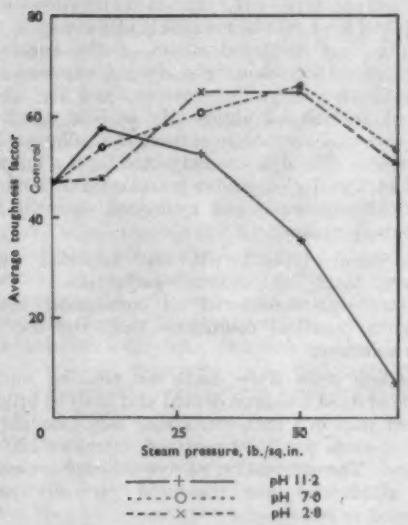

FIG. 1—Effect of High Temperatures on Terylene Yarn at Various pH Values

Under neutral or acid conditions the fibre withstood the temperature satisfactorily, actually increasing in toughness up to a steam pressure of 50 lb./sq.in. Under mildly alkaline conditions, however, degradation occurred rapidly at pressures over 30 lb./sq.in. This means that efficient rinsing must follow scouring where dyeing is carried out above 100°C., particularly if sodium carbonate or caustic soda is used. As a safeguard an acetic acid rinse may be given to ensure that the last traces of alkali are eliminated from the fibre.

2. DYE STABILITY

Table I shows—(i) the wavelength of maximum absorption (λ_{max}) of the Dispersol and Duranol dyes examined; in some cases two maxima were

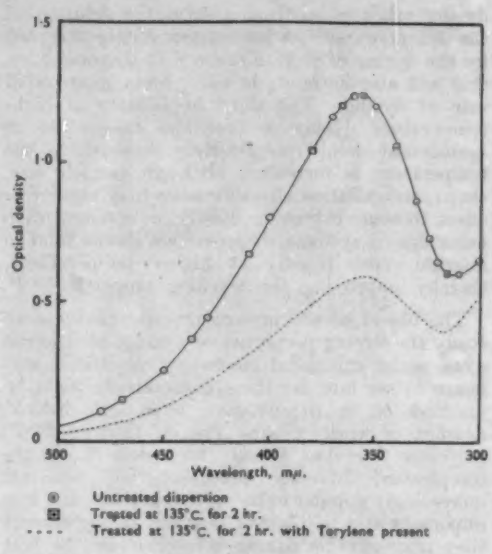

FIG. 2—Stability of Dispersol Fast Yellow A (Class (*a*)) at 135°C.

present; (ii) the optical density at λ_{max} (d) of the standard solution in methanol before and after heating; and (iii) the "stability class" defined as follows—

(*a*) Less than 5% decomposition, based on the values of d

(*b*) More than 5% decomposition, but curves similar in shape

(*c*) Curves before and after heating dissimilar.

Fig. 2–4 illustrate the behaviour under these conditions of selected dyes from each of the classes (*a*), (*b*), and (*c*).

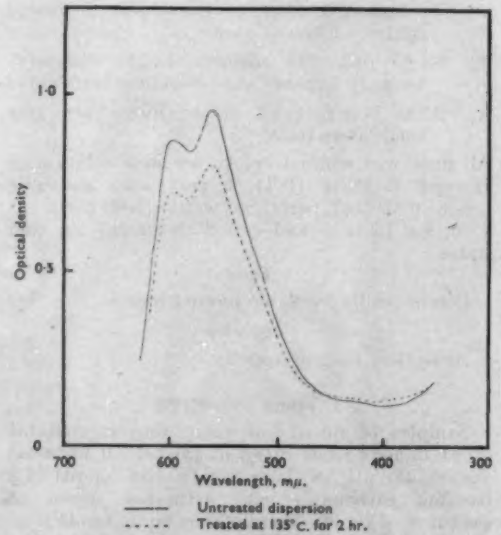

FIG. 3—Stability of Duranol Violet RN (Class (*b*)) at 135°C.

TABLE I

Effect of Heat Treatment at 135°C. on Stability of Disperse Dyes

Dye	λ_{max} (mμ.)	Optical Density at λ_{max} Before Heating	Optical Density at λ_{max} After Heating	Class
Duranol Brilliant Yellow 6G ...	434	0·230	0·230	*a*
Dispersol Fast Yellow A	357	0·620	0·610	*a*
Dispersol Fast Yellow G	356 395	0·800 0·610	0·755 0·585	*a*
Dispersol Fast Yellow GR	378	0·515	0·485	*b*
Duranol Orange G	480	0·205	0·220	*a*
Dispersol Fast Orange A	475	0·515	0·525	*a*
Dispersol Fast Orange B	433	0·310	0·285	*b*
Dispersol Fast Orange G	440	1·060	1·050	*a*
Dispersol Fast Orange Brown RN ...	443	1·195	1·150	*a*
Duranol Red GN	510	0·990	0·935	*b*
Duranol Red 2B	530 565	0·860 0·700	0·820 0·670	*a*
Duranol Red X3B	570 530	1·070 1·000	1·055 0·990	*a*
Dispersol Fast Red R	478	0·710	0·690	*a*
Dispersol Fast Scarlet B	485	1·230	1·170	*a*
Dispersol Fast Crimson B	503	1·320	1·300	*a*
Duranol Violet 2R	545 585	1·820 1·670	1·835 1·690	*a*
Duranol Violet RN	564 597	0·950 0·865	0·800 0·720	*b*
Duranol Brilliant Violet BR ...	563 605	1·690 1·660	1·630 1·620	*a*
Duranol Blue G	—	—	—	*c*
Duranol Blue GN	—	—	—	*c*
Duranol Blue Green B	—	—	—	*c*

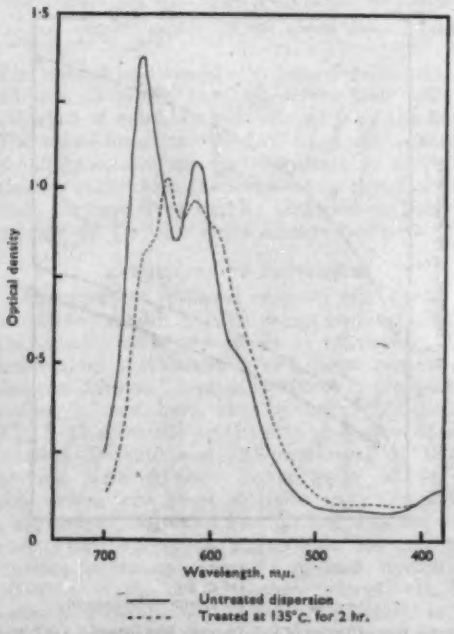

FIG. 4— Stability of Duranol Blue Green B (Class (*a*)) at 135°C.

The majority of dyes showed no appreciable decomposition under these severe conditions. Dispersol Fast Yellow GR, Dispersol Fast Orange B, Duranol Red GN, and Duranol Violet RN decomposed to a slight extent. The absorption curves of the three blue dyes underwent profound changes, but when dyeings were carried out with the heated dispersion of Duranol Blue G at 100°C. (in the presence of *o*-phenylphenol as assistant) no significant difference in colour or in fastness properties was observed, in comparison with a dyeing from the unheated control dispersion. The optical absorption technique is therefore a very sensitive method of detecting structural changes, and it may be safely concluded that no serious difficulties will arise with dye decomposition up to 130°C. for 2 hr.

3. EFFECT OF TEMPERATURE

The advantage of using temperatures above 100°C. under practical dyeing conditions was shown in an early experiment in which Dispersol Fast Scarlet B and Duranol Blue Green B were applied to Terylene at temperatures from 85° to 130°C. for 45 min., at a liquor : goods ratio of 20 : 1. Fig. 5 clearly shows the continuous improvement in dyebath exhaustion as the temperature is raised.

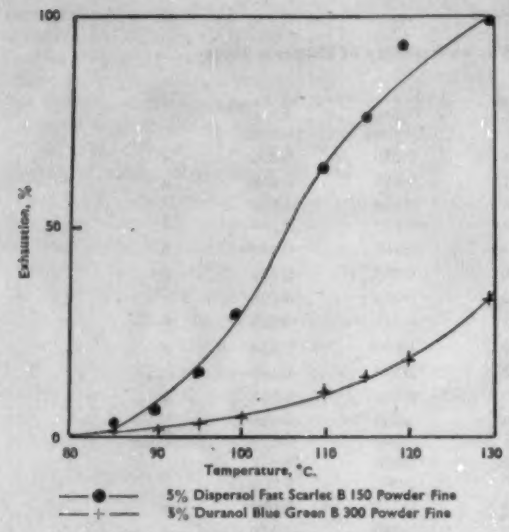

FIG. 5— Effect of Temperature on Exhaustion after 90 min.

A more detailed examination of Dispersol Fast Scarlet B was then made. Fig. 6 shows the amount of dye absorbed by Terylene from dyebaths containing various amounts of dye after 90 min. at 85°, 100°, and 120°C. (all in absence of assistant), at a liquor ratio of 30 : 1. At 120°C. the dye absorbed increases linearly with the initial dyebath concentration, whereas at 85° and 100°C. the fibre appears to be saturated at an early stage. The rate-controlling step of the dyeing process is probably the diffusion of dye from a saturated layer on the fibre surface; at the lower temperatures this layer becomes saturated at an initial dyebath concentration of 2–4%, and as the rate of diffusion into the fibre is so low, no more dye can be absorbed in this time at higher initial dyebath concentrations. At 120°C., however, dye diffuses rapidly into the fibre, and the surface layer can be continually replenished by dye from the bath.

Rate-of-dyeing experiments were next carried out with two dyes of widely different dyeing rates on Terylene— Duranol Red 2B (quickly diffusing)

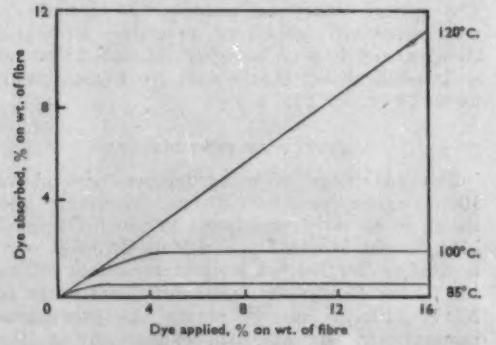

FIG. 6— Effect of Temperature on Absorption of Dispersol Fast Scarlet B 150 Powder Fine

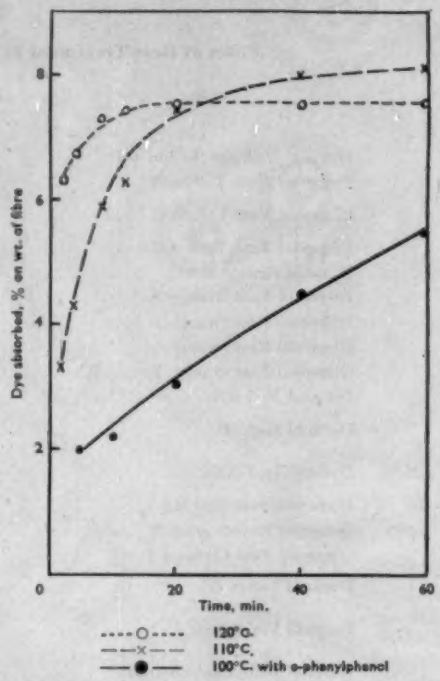

FIG. 7— Rate of Dyeing of 10% Duranol Red 2B 300 Powder Fine

and Dispersol Fast Orange B (slowly diffusing), at 110°C. and 120°C., and at 100°C. in presence of *o*-phenylphenol (3 g./litre) as assistant. The results are illustrated in Fig. 7 and 8.

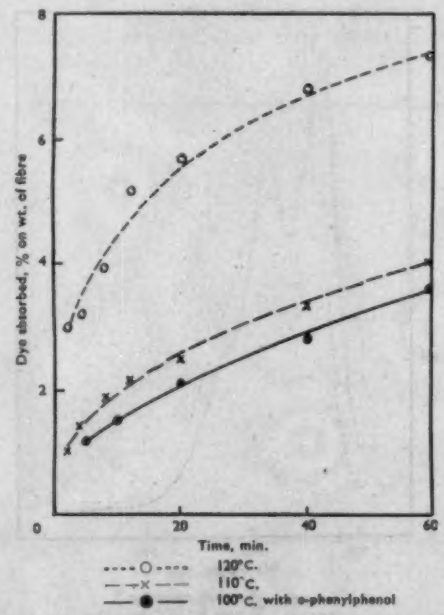

FIG. 8— Rate of Dyeing of 10% Dispersol Fast Orange B 300 Powder Fine

Duranol Red 2B clearly reaches equilibrium under these conditions in 20 min. at 120°C. and in about 60 min. at 110°C., but does not reach equilibrium after 60 min. at 100°C. The curves at 120°C. and 110°C. cross in the expected manner, and show that for quickly diffusing dyes an increase in the dyeing temperature may speed up the initial dyeing rate but reduce the dye uptake at equilibrium. Dispersol Fast Orange B, on the other hand, does not reach equilibrium after 60 min. even at 120°C., but the differences in the amounts of dye taken up after, say, 30 min. at each temperature (viz. 2·25, 2·75, and 6·0% in ascending order) illustrate the great increase in dyeing rate conferred by the rise in temperature from 110° to 120°C. With such dyes, a temperature of at least 130°C. will be required for equilibrium to be reached in 60 min. This increase in temperature will, however, cause a marked reduction in the exhaustion of the more quickly diffusing dyes. For economic reasons, therefore, it will probably be preferable, with existing disperse dyes, to restrict the dyeing temperature to 120°C., and to accept the slight inefficiency shown by the most slowly diffusing dyes at this temperature.

It is interesting to compare at this stage the times of half-dyeing of these dyes on Terylene at 120°C. and on acetate rayon at 85°C. (Table II).

TABLE II

Times of Half-dyeing

($t_{\frac{1}{2}}$ minutes)

	Acetate Rayon at 85°C.	Terylene at 100°C.	Terylene at 120°C.
Duranol Red 2B ...	2·4	>15	<2
Dispersol Fast Orange B	19	>15	5–12*

* The uncertainty in these figures arises because the equilibrium uptake is not known for certain, but it must lie between 10% (the amount originally applied) and the amount actually absorbed after 60 min.

The time of half-dyeing $t_{\frac{1}{2}}$ is defined as the time in minutes at which half the dye which will be absorbed at equilibrium is taken up by the fibre. The values for acetate rayon [17] have only a limited significance, and are quoted merely to illustrate that, provided that the temperature is sufficiently high, disperse dyes can be absorbed at least as quickly by Terylene as by acetate rayon at 85°C.

4. BUILDING-UP PROPERTIES

An important practical property of a dye is its ability to produce heavy dyeings economically as the amount of dye applied is increased. The building-up characteristics of a large number of disperse dyes on Terylene at 100°C. in presence of *o*-phenylphenol have already been published [18]. Fig. 9–13 show the building-up properties of five selected Duranol and Dispersol dyes at 120°C. (dyeing time 60 min., liquor ratio 40 : 1), in comparison with those at 100°C. from the earlier publication. Dispersol Fast Yellow A is the only case where dyeing at 120°C. does not confer a distinct advantage in build-up over dyeing at 100°C. with the aid of *o*-phenylphenol. In the other four cases (and this applies generally to all the dyes examined) it is possible to obtain depths at 120°C. which are not obtainable at 100°C. Furthermore, in many cases the degree of exhaustion is high enough, even at 10–15% depths, to enable these very heavy dyeings to be dyed economically at 120°C.

Table III shows all the dyes in order of their building-up properties as measured by the degree of exhaustion over the range 0–10% of applied dye under the standard conditions described above.

TABLE III

Building-up Properties of Disperse Dyes on Terylene at 120°C.

70–100% EXHAUSTION— GOOD BUILD-UP
- Duranol Brilliant Yellow 6G
- Dispersol Fast Yellow GR
- Duranol Orange G
- Dispersol Fast Orange A
- Dispersol Fast Orange B
- Dispersol Fast Orange Brown RN
- Dispersol Fast Scarlet B
- Duranol Red GN
- Duranol Red 2B
- Dispersol Fast Crimson B
- Duranol Blue G
- Duranol Blue GN

40–70% EXHAUSTION— MODERATE BUILD-UP
- Dispersol Fast Yellow A
- Dispersol Fast Yellow G
- Dispersol Fast Orange G
- Duranol Red X3B
- Duranol Violet RN
- Duranol Violet 2R
- Duranol Brilliant Violet BR

0–40% EXHAUSTION— POOR BUILD-UP
- Dispersol Fast Red R
- Duranol Blue Green B

The effect of dyebath assistants like Tumescal D and *o*-phenylphenol (2-hydroxydiphenyl) on the build-up of disperse dyes is so marked at 100°C. that their behaviour was studied at 120°C. to discover whether a similar advantage could be obtained. Dispersol Fast Yellow G (quickly diffusing), Duranol Brilliant Violet BR, and Duranol Blue Green B (both slowly diffusing) were

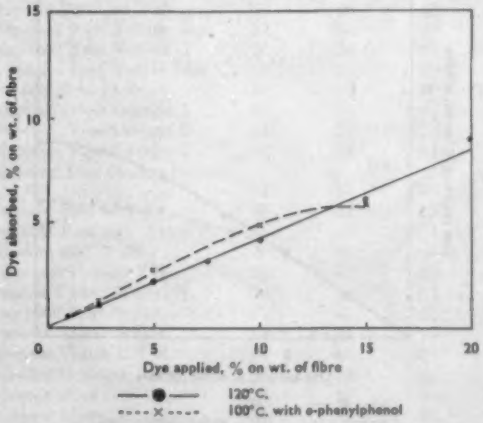

FIG. 9— Build-up of Dispersol Fast Yellow A 300 Powder Fine

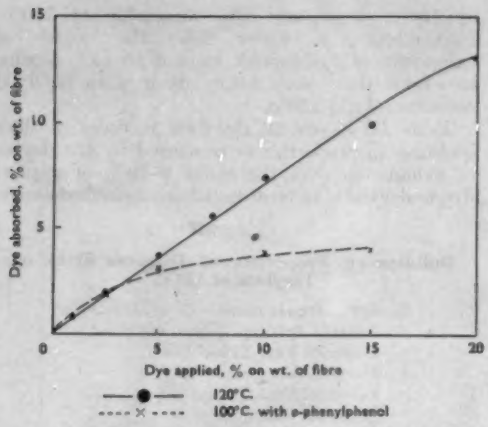

FIG. 10— Build-up of Dispersol Fast Orange B 300 Powder Fine

dyed at 120°C. for 60 min. in absence of assistant and in presence of *o*-phenylphenol (3 g. per litre of dye liquor). The results are illustrated in Fig. 14–16. With Dispersol Fast Yellow G and Duranol Blue Green B there is a significant improvement in the amount of dye taken up when *o*-phenylphenol is present, except at the highest concentration employed with Dispersol Fast Yellow G. The absorption of Duranol Brilliant Violet BR, on the other hand, is not appreciably affected by this assistant. These results show that dyes can vary widely in their sensitivity to *o*-phenylphenol at 120°C., and that this cannot be easily correlated with the relative rates of diffusion of the dyes into Terylene. It seems as if, with certain dyes, *o*-phenylphenol can increase the volume of fibre through which dye can readily pass, since with Dispersol Fast Yellow G, in particular, equilibrium will easily be reached at 120°C. after 60 min. dyeing and the effect of *o*-phenylphenol cannot be exclusively due to an increased rate of dyeing.

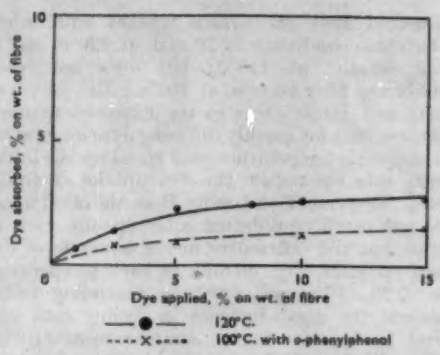

FIG. 12— Build-up of Duranol Brilliant Violet BR 300 Powder Fine

5. EFFECT OF pH

Variations in dyebath acidity can readily arise under practical conditions for a variety of reasons, and it is essential to know how these can affect the amount of dye absorbed. The behaviour of two dyes, Dispersol Fast Yellow A and Dispersol Fast Orange B, is shown in Fig. 17, in which the amount of dye taken up under standard conditions of temperature, time, and liquor ratio is plotted as a function of the initial dyebath pH. The uptake of Dispersol Fast Orange B is unaffected by variations in pH over the range 3·2–9·2, although a slight falling-off is observed in more alkaline baths. In contrast, Dispersol Fast Yellow A is continuously sensitive to dyebath acidity in the pH range 3·3–8·6, the amount of dye absorbed falling off rapidly above about pH 6·0. As a precaution, therefore, sufficient control must be applied to disperse dyebaths for Terylene to ensure that large variations in pH cannot occur during dyeing. In particular, softened waters containing bicarbonates, which increase in alkalinity on boiling, must be efficiently neutralised, and all Terylene fibre must be efficiently washed off after scouring with alkali, before being dyed at high temperatures.

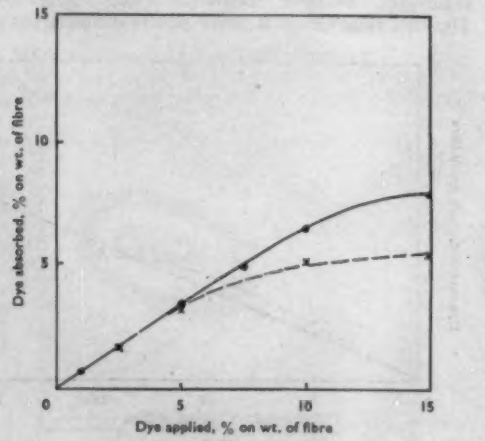

FIG. 11— Build-up of Duranol Red X3B 300 Powder Fine

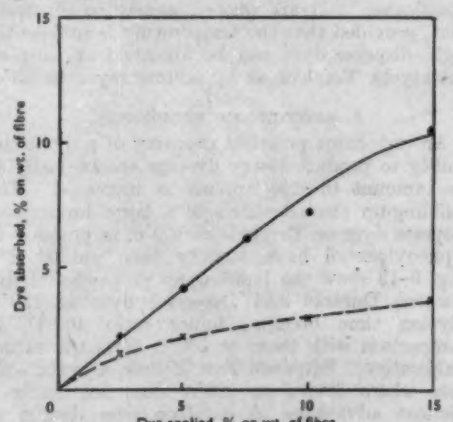

FIG. 13— Build-up of Duranol Blue G 300 Powder Fine

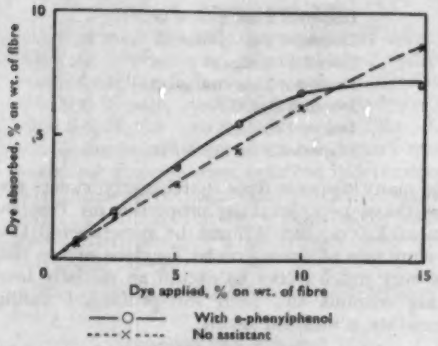

FIG. 14—Dispersol Fast Yellow G 300 Powder Fine—Effect of Assistant on Uptake at 120°C.

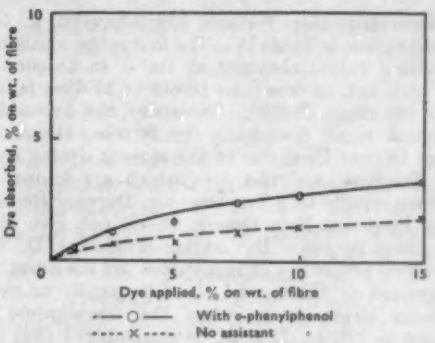

FIG. 16—Duranol Blue Green B 300 Powder Fine—Effect of Assistant on Uptake at 120°C.

Dispersol Fast Yellow A, Dispersol Fast Yellow GR, and Duranol Blue Green B are the only dyes of those examined which are sensitive to large changes in pH, under practical dyeing conditions. Although there is no great problem if the dyebath pH changes accidentally, it is also clear that no advantage is to be gained by deliberately adding acid or alkali to disperse dyebaths in order to promote the exhaustion on Terylene.

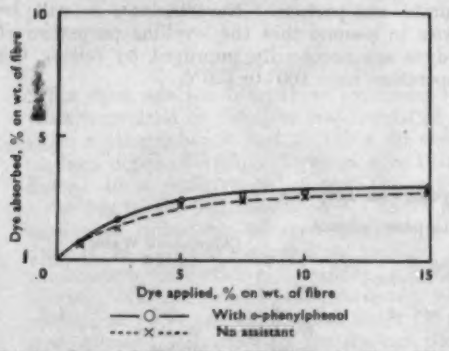

FIG. 15—Duranol Brilliant Violet BR 300 Powder Fine—Effect of Assistant on Uptake at 120°C.

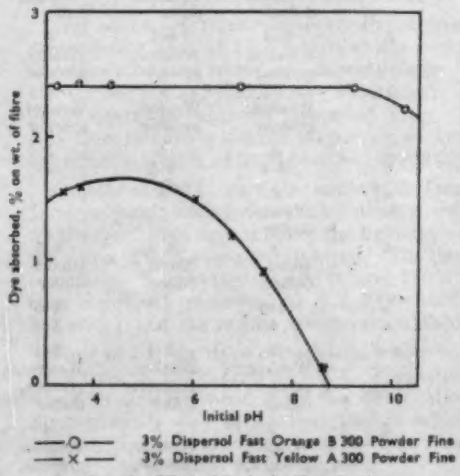

FIG. 17—Effect of Dyebath pH on Uptake

6. LEVELLING

It is a property of all known dyeing systems that the rate of levelling, i.e. the transfer of dyes from more heavily dyed portions to less heavily dyed portions, increases as the temperature rises [14].

This may simply be regarded as a result of the increased rate of diffusion of the dye in both aqueous and fibre phases, but for dyes which reach equilibrium within the time used for levelling, it may also be connected with the tendency, as the temperature rises, for the exhaustion at equilibrium to decrease and consequently for the amount of levelling to increase. This phenomenon is well known for acid dyes on wool [19].

The conventional white-skein levelling test, previously used for direct dyes on cotton by Lemin, Vickers, and Vickerstaff [20], was used in this work, and the levelling for each dye, under conditions described in the previous section, is given in Table IV.

TABLE IV

Levelling of Disperse Dyes on Terylene at 100°C. and 120°C.

(Liquor ratio 30 : 1)

Dye (3% on wt. of material)	Levelling (%) (*a*) 100°C. 90 min.	(*b*) 100°C.+ Tumescal D 90 min.	(*c*) 120°C. 60 min.
Duranol Brilliant Yellow 6G	21	26	40
Dispersol Fast Yellow A	33	57	79
Dispersol Fast Yellow G	30	70	77
Dispersol Fast Yellow GR	32	32	40
Duranol Orange G ...	24	86	99
Dispersol Fast Orange A	23	14	34
Dispersol Fast Orange B	39	27	16
Dispersol Fast Orange G	25	72	91
Dispersol Fast Orange Brown RN	22	33	50
Duranol Red GN ...	28	52	73
Duranol Red 2B ...	28	57	84
Duranol Red X3B ...	28	66	64
Dispersol Fast Red R	25	57	50
Dispersol Fast Scarlet B	26	45	71
Dispersol Fast Crimson B	23	32	49
Duranol Violet 2R ...	28	51	66
Duranol Violet RN ...	25	45	48
Duranol Brilliant Violet BR	26	52	45
Duranol Blue G ...	28	35	39
Duranol Blue GN ...	24	36	36
Duranol Blue Green B	25	42	39

Two surprising features are observed in the results given in Table IV. The first is the consistent levelling values obtained at 100°C. in absence of an assistant, no less than 18 out of 21 dyes falling into the range 21–30%. Secondly, the dye which levelled most completely in 90 min. (Dispersol Fast Orange B) is one of the slowest-dyeing dyes on Terylene, and the dyes which are known to diffuse rapidly into Terylene, e.g. Duranol Red 2B and Dispersol Fast Orange G, do not give high levelling figures. By adding Tumescal D, the levelling properties of many dyes are considerably improved at 100°C., and there is usually an even greater improvement when the temperature is raised to 120°C. It must be remembered that the depth of the dyeing on which the levelling test is carried out is much less in column (*a*) than in columns (*b*) and (*c*).

If a dye of satisfactory levelling properties is defined as one which gives a figure of 70% migration under the conditions specified above, none of the dyes examined is satisfactory at 100°C. in absence of Tumescal D. The following are satisfactory at 100°C. in presence of this assistant—

Dispersol Fast Yellow G
Duranol Orange G
Dispersol Fast Orange G

The number of satisfactory dyes is greater at 120°C.—

Dispersol Fast Yellow G
Dispersol Fast Yellow A
Duranol Orange G
Dispersol Fast Orange G
Duranol Red GN
Duranol Red 2B
Dispersol Fast Scarlet B

Thus many disperse dyes, particularly violets and blues, have poor levelling properties on Terylene even at 120°C., but it must be remembered that the slow rate of dyeing on to Terylene means that it is very much easier to obtain an initially level dyeing without the need for prolonged boiling to produce a uniform result.

Three dyes—

Dispersol Fast Orange B
Dispersol Fast Red R
Duranol Brilliant Violet BR

show less levelling at 120°C. than at 100°C. in presence of Tumescal D. There is insufficient fundamental knowledge at present available to discuss the reason for this, and in any case the conditions under which the tests were made at the two temperatures were too dissimilar to permit an accurate comparison. Nevertheless, it will be unwise to assume that the levelling properties of all dyes are necessarily improved by raising the temperature from 100° to 120°C.

TABLE V

Wet Fastness of Disperse Dyeings on Terylene

Dye	Dyeing Method	(*a*) 120°C. (*b*) 100°C. with *o*-phenylphenol 1 Wash at 85°C. Effect on Dyeing	Staining of Wool	Staining of Terylene	Chlorinated Water pH 6	pH 7	pH 8
Dispersol Fast Yellow A ...	(*a*)	5	5	5	4 Weaker, duller	4–5 Weaker, duller	4–5
	(*b*)	4	4–5	5	3 Weaker, duller	4 Weaker, duller	4 Weaker, duller
Dispersol Fast Yellow G ...	(*a*)	5	4–5	5	3–4 Weaker, greener	4–5 Weaker, greener	4 Weaker, greener
	(*b*)	4	4–5	5	2 Weaker, greener	3 Weaker, greener	3 Weaker, greener
Dispersol Fast Orange B ...	(*a*)	5	4–5	5	4	5	5
	(*b*)	3–4	4	5	3–4	4–5	4
Duranol Red 2B	(*a*)	5	4–5	5	4 Bluer, duller	4	5
	(*b*)	3–4	3–4	4	4 Bluer, duller	4–5 Bluer, duller	4–5 Bluer, duller
Duranol Red X3B ...	(*a*)	5	4	5	4–5	4–5	4–5
	(*b*)	4	3–4	4	4	4	4–5
Duranol Blue G	(*a*)	4–5	4–5	4–5	3 Weaker, greener, duller	3–4 Weaker, greener, duller	3–4 Weaker, greener, duller
	(*b*)	4–5	4–5	5	3 Weaker, greener, duller	3 Weaker, greener, duller	4 Weaker, greener, duller

7. COMPATIBILITY

A dye must not only be capable of building up "on tone": when used in combination with other dyes of similar fastness, the same hue should be obtained in pale, medium, and heavy dyeings—in other words, the dyes must be compatible. Schuler and Remington [12] showed that in binary mixtures dissimilar disperse dyes behaved independently of each other on polyethylene terephthalate yarn at 90°C., although closely related dyes exhibited some interference, as do certain acid dyes on polyamide fibres.

The compatibility of binary and ternary mixtures of four dyes—

Dispersol Fast Yellow A
Dispersol Fast Orange B
Duranol Red X3B
Duranol Blue G

was examined on Terylene staple yarn at 120°C. These dyes were selected because they are already widely used in combination for mode shades having the best light and sublimation fastness, consistent with reasonably good dyeing properties. Fig. 18 shows the results obtained. In no case is there any evidence of the preferential absorption of one or more dyes, and it seems safe to assume that incompatibility will not occur in this type of dyeing system.

8. FASTNESS PROPERTIES

Six dyes selected from those examined in this work were dyed on Terylene staple yarn at 100°C. (with *o*-phenylphenol) and at 120°C. in the same medium depth of colour. Dyeings were then submitted to a wide range of wet fastness tests according to the ICI handbook [21]. There was no significant difference between the two sets of dyeings in fastness to alkaline milling, water, chlorination, carbonising, decatising, potting, and stoving. Certain severe tests, however, such as washing at 85°C. and chlorinated water (25 p.p.m. free chlorine), indicate that the dyeings obtained at 120°C. are slightly superior in fastness. The detailed comparisons are shown in Table V.

This slight superiority supports the supposition made earlier that at high temperatures regions of the fibre structure which are inaccessible to dye at 100°C. become available for absorption. Any low-temperature aqueous treatment after dyeing may then remove a smaller proportion of dye, but the effect is clearly of little practical importance.

Dyeings at 100°C. were also made with Tumescal D as assistant, since traces of this product retained by the dyed fibre do not affect the light fastness to the same extent as *o*-phenylphenol. The daylight fastness of the dyeings at 100°C. and 120°C. were then compared (according to *B.S.* 1006 : 1953 on a 1–8 scale), and the results are shown in Table VI.

Four of the six dyes are slightly faster to light when dyed at 120°C. than at 100°C. This tendency, which is in agreement with the observations of Zimmerman [1], may be another way in which the superior penetration of dyes into the fibre structure at high temperature manifests itself.

TABLE VI

Light Fastness of Disperse Dyes on Terylene

(*a*) 120°C. (*c*) 100°C. with Tumescal D

Dyeing method ...	(*c*)	(*a*)
Dispersol Fast Yellow A ...	6–7	6–7
Dispersol Fast Yellow G ...	6–7	7
Dispersol Fast Orange B ...	6–7	7
Dispersol Fast Red 2B ...	6 duller	6 duller
Duranol Red X3B	5	5–6
Duranol Blue G	5 redder	6 redder

Conclusions

(1) Terylene fibre can be treated for 2 hr. at temperatures up to 160°C. in the pH range 2·8–7·0 without a reduction in its toughness factor. If the dyebath is allowed to become alkaline, degradation may set in rapidly at temperatures above 100°C.

(2) The optical absorption curves of most disperse dyes are unaffected by heating at 130°C. for 2 hr. Even where the optical absorption characteristics of the dye change appreciably as a result of this treatment, i.e. with the three blue dyes examined, there is still no apparent change in the colour and fastness properties of the derived dyeings. Dye instability should not, therefore, present a problem in bulk high-temperature dyeing.

(3) With existing Duranol and Dispersol dyes, no advantage is to be gained by dyeing Terylene at temperatures much above 120°C. Although superior results may be obtained with very slow-dyeing dyes at 130°C., the reduction in exhaustion of the remainder means that this higher temperature is relatively uneconomic. At 120°C., the times of half-dyeing of most disperse dyes on Terylene are comparable with those of the same dyes on acetate rayon at 85°C.

(4) The large majority of dyes possess moderate to good exhaustion in 60 min. at 120°C., and can be used economically for even the heaviest of dyeings. With the exception of Dispersol Fast Yellow A, the building-up properties of disperse dyes on Terylene at 120°C. are much superior to those at 100°C. in presence of *o*-phenylphenol as assistant. This means that colours can be obtained at 120°C. which are unobtainable, as far as depth is concerned, at 100°C.

(5) The disperse dyes examined vary in their sensitivity at 120°C. to assistants such as *o*-phenylphenol, and the influence of this agent on building-up properties does not seem connected with the rate of diffusion of the dye. The indiscriminate use of dyeing assistants at temperatures above 100°C. cannot be justified at the present time.

(6) Certain dyes are very sensitive to alkaline dyebath conditions. No advantage is gained by adding acid deliberately to the dyebath, and dyeing with disperse dyes should therefore be carried out under substantially neutral conditions. Care must be taken to neutralise any compounds in the water which may become alkaline on prolonged treatment at 120°C.

(7) The levelling properties of many dyes are considerably improved at 120°C., although the violet and blue members of the range have poor levelling properties even at this temperature.

(8) There is no evidence of dye interference on the fibre in binary and ternary mixtures of four widely used disperse dyes. Incompatibility difficulties should not exist when dyeing Terylene at 120°C.

(9) Dyeings carried out at 120°C. are slightly faster to certain wet treatments and to light than those at the same depth carried out at 100°C. No reduction–clearing treatment is necessary for the majority of pale, medium, and heavy depths if dyeing is carried out at 120°C., provided that sufficient time has been allowed for dye penetration to take place.

(10) If Terylene is dyed at 120°C. a high degree of exhaustion is obtained from most disperse dyes in a practical dyeing time (1 hr. or less), expensive carriers are unnecessary, and in many cases clearing treatments after dyeing can be eliminated. High-temperature techniques should therefore be preferred to dyeing at 100°C. with a carrier, provided that other economic factors permit, and that suitable equipment for processing the fibre, yarn, or fabric is available.

* * *

The practical work described in this paper was carried out by Mr. A. Morrison.

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(*MS. received 11th March 1955*)

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Discussion

Mr. F. M. STEVENSON: The bulk of the work reported tonight has been carried out in the region 120–130°C., but it is anticipated that much higher temperatures might give even better results. Since little work appears to have been done on this point, is there some limiting factor that has not emerged in the paper?

Mr. FERN: The present investigation shows that there is no advantage with the existing disperse dyes in dyeing above about 130°C. Novel dyes might well be designed which are applicable only at temperatures of 130–200°C.; these would probably possess much better wet and sublimation fastness than the present types. Confirmation of this trend is given by the recent successful application of Brenthol OT (ICI) to Terylene at 130°C., providing the basis of a one-stage process for fast blacks. Limiting factors are fibre stability, dye stability, and the ability of the equipment to withstand high pressures.

Mr. W. PICKFORD: Has the lecturer any information on the use of methyl salicylate as a carrier?

Mr. FERN: Methyl salicylate is an effective carrier for disperse dyes on Terylene. It is, however, retained very tenaciously by the fibre, and the resulting smell is thought by many to be a severe disadvantage.

Mr. J. C. HAWLEY: How could a temperature of 200°C. be obtained in bulk practice?

Mr. FERN: There is no theoretical or practical difficulty in designing a dyeing machine to work at 200°C. (220 lb./sq. in. saturated steam). Pressures much higher than this are encountered daily in other industries.

Mr. HAWLEY: Has any work been done on the behaviour of acid dyes at high temperatures?

Mr. FERN: Acid dyes are generally too soluble in water to possess much substantivity for Terylene, and their absorption is not increased by raising the temperature to 130°C.

Mr. STEVENSON: Would the lecturer care to speculate on the spin-dyeing of Terylene in the future?

Mr. FERN: The only technical problem connected with spun-dyed Terylene is the discovery of a sufficiently wide range of colouring matters, of suitable fastness, which are stable to the spinning conditions involved. Once this problem is solved, the production of spun-dyed yarn depends only on commercial considerations.

Mr. H. W. PARTRIDGE: Can information be given on "dry-heat" dyeing. I understand that this has already been tried in printing?

Mr. FERN: The "dry-heat" or Thermosol (DuP) process has been used successfully on the small scale to dye Terylene piece goods continuously, and it has limited possibilities in the dyeing of loose staple and slubbing. The material is padded with a dispersion of a suitable disperse or vat dye, or a pigment, dried, and heated at

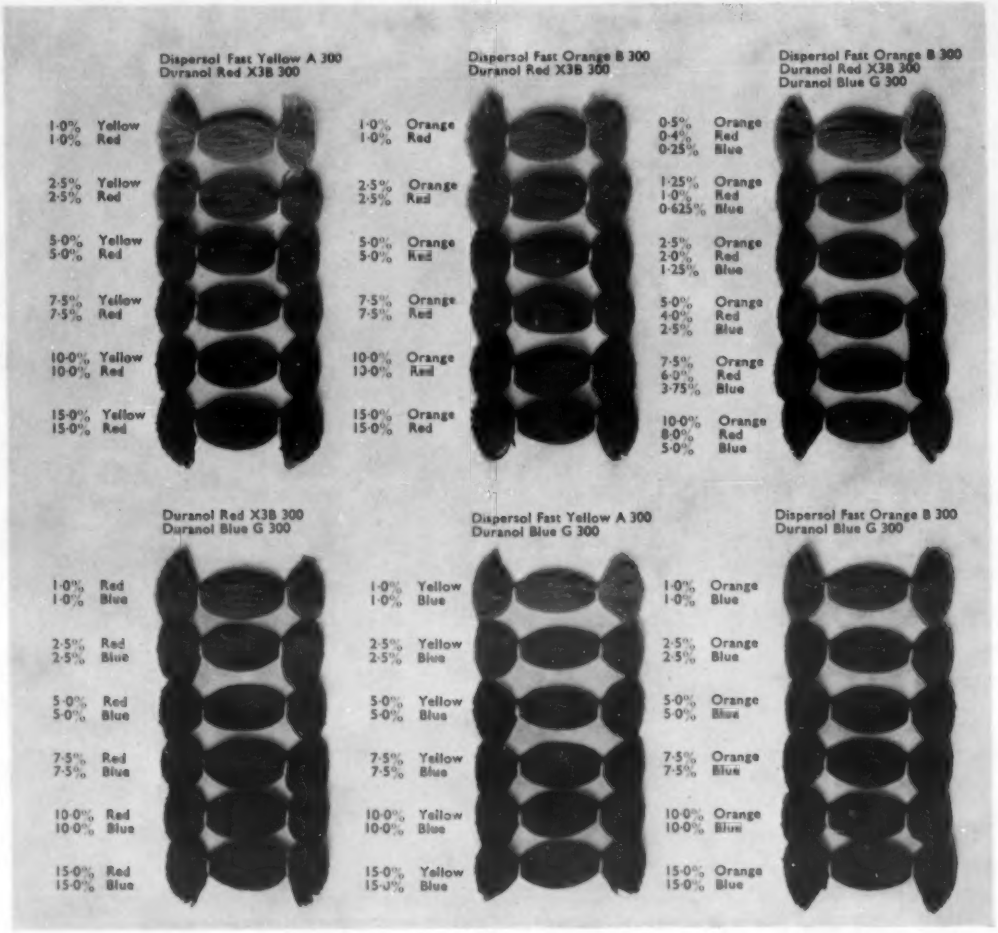

FIG. 18—Compatibility of Disperse Dyes on Terylene at 120°C.

200–230°C. for 5–30 sec., whereupon dyeing takes place. A reduction–clearing treatment concludes the process. The assembly of equipment required for the successful application of this technique in bulk is probably unavailable in the United Kingdom, and the process has so far not aroused much interest.

Mr. PARTRIDGE: It has been stated elsewhere that a few disperse dyes on Terylene behave anomalously in that light fastness decreases with depth of dyeing. Is this so?

Mr. FERN: Certain dyes do, indeed, undergo a reduction in daylight fastness on Terylene as the depth is increased. This applies to dyeings made at 100° or 120°C. Examples are—

Duranol Orange G
Duranol Red GN
Duranol Violet 2R
Duranol Brilliant Violet BR
Duranol Brilliant Violet B
Duranol Blue 2G
Duranol Blue GN
Duranol Brilliant Blue G
Duranol Brilliant Blue B

No explanation for this phenomenon can be advanced.

Mr. S. R. MEADOWS: Has any work been done on high-temperature union dyeing of Terylene and wool?

Mr. FERN: Laboratory work is in progress on this topic, and some preliminary results are described by Fern and Hadfield [22].

Mr. B. KRAMRISCH: Is there any material difference in rubbing fastness between dyeings of equal depth produced by the carrier and by the high-temperature techniques?

Mr. FERN: In a given dyeing system there will usually be a depth of colour beyond which the reduction–clearing process must be given in order to obtain dyed Terylene which exhibits good rubbing fastness. Generally, this depth will be considerably greater for dyeings at 120°C. than for dyeings at 100°C. with a carrier.

Mr. KRAMRISCH: Has any difference in rubbing fastness been observed when the same dye is applied to filament and staple Terylene?

Mr. FERN: Although there is no direct evidence, it is thought that any differences in rubbing fastness before clearing will be associated more with variations in dyeing temperature and time, and the form in which the Terylene is dyed (i.e. yarn, fabric, etc.), rather than with the variation between staple and filament Terylene.

Wear Properties of Resin-finished Rayon Staple Fabrics— A New Method of Laboratory Assessment

L. F. H. BREENS and T. H. MORTON

Meetings of the London Section held in the rooms of the Royal Society, Burlington House, London W.1, on 1st October 1954, Mr. R. A. McFarlane in the chair; and of the Manchester Section held at the Textile Institute, Manchester, on 17th December 1954, Mr. G. S. J. White in the chair

An analysis of the problem of the prediction of premature breakdown of crease-resist-finished pure and blended rayon staple fabrics in heavy-duty wear shows that current methods of laboratory assessment, based on flat abrasion motions, are inadequate. A new type of testing machine, the BFT Mark III, determines resistance to flexing and to ball penetration; from these two machine factors a "duty factor" is derived which correlates very well with resistance to premature wear in a series of about sixty fabrics whose wear behaviour is known with certainty. Three different mechanisms of abrasive breakdown of woven fabric are distinguished and related to measurable machine factors; two only of these, ball penetration and flex abrasion, are components of the duty factor.

1. INTRODUCTION

In physical science, the first essential step in the direction of learning any subject is to find the principle of numerical reckoning and practicable methods for measuring some quality connected with it. I often say that when you can measure what you are speaking about and express it in numbers you know something about it; but when you cannot measure it, when you cannot express it in numbers, your knowledge is of a meagre and unsatisfactory kind: it may be the beginning of knowledge, but you have scarcely, in your thoughts, advanced to the stage of science, whatever the matter may be.—*Lord Kelvin* [1].

It is generally recognised by textile finishers and by the textile trade that the wrong application to pure and blended rayon staple of certain finishes based on urea–formaldehyde and melamine –formaldehyde resins can result in the production of poor-wearing fabrics. A very large amount of dress-weight, crease-resist-finished rayon staple has been produced, and the proportion of consumer complaints has been very small. However, with the introduction of rayon staple cloths into the heavy-duty apparel field the proportion of complaints has become larger, and it is generally believed that these poor-wearing fabrics constitute an important trade problem.

It is true to say that the majority of fabrics made from rayon staple and given a resin finish are satisfactory; but the residuum of unsatisfactory cloths has proved difficult to deal with,

for it is the experience of the finishing trade that there is no satisfactory laboratory method of diagnosing a poor-wearing fabric. Normal chemical testing for resin content and resin stability is of no use as a diagnostic method; further, tensile testing is of little use insofar as only a few of the fabrics which are the subject of complaint show abnormally low strength or extensibility.

Since the complaints are of wear, it has been customary to test in an abrasion machine of one type or another. All the machines commonly used in this country for this purpose can be classified as flat abraders; all seem to be imperfect diagnostic instruments, for, while they may show that a proportion of the cloths complained of have low abrasion-resistance, they also indicate that the remainder have a relatively high abrasion-resistance. Our experiments confirm that flat-abrasion machines, in general, are not a satisfactory method of predicting poor wear in a resin-finished rayon staple fabric.

So far as can be ascertained, laboratories in this country charged with the routine examination of resin-treated fabrics use variously Linra, Martindale, Ring-Wear[2], and Wyzenbeek machines; corresponding laboratories in the United States generally use Schiefer, Stoll, Taber, and Wyzenbeek abraders[3].

It is indicated above that current abrasion machines are imperfect in prediction of poor wear. Since any kind of wear is essentially a deterioration of the fabric by mechanical action, it is reasonable to expect that machines can be devised which will reproduce basic wear mechanisms: it has been our purpose to devise machine methods which will give satisfactory wear predictions. We have been encouraged in this course by the observations of a number of workers, including J. C. Mann[4] of this laboratory, that fabrics may be ranked in different orders by abrasion machines of differing mechanism. It has been our aim to devise a series of mechanisms which will provide machine data to be correlated with serviceability. This paper describes some of the mechanisms we have devised and their application to the assessment of the performances of resin-finished rayon staple fabrics.

The discussion on the correlation of wear properties with abrasion machine results is confined to fabrics of rayon staple and its blends in heavy-duty use, i.e. in slacks, men's suits, uniforms, shirts, overalls, gym-slips, and raincoats. It is our experience that light-duty fabrics, e.g. dress materials and women's suitings, do not constitute a significant wear problem, although a study of such fabrics has been essential in reaching our conclusions.

We have examined a large number of garments which have broken down prematurely in use, and find that the short wear-life is due to a particular kind of damage, which may be termed *erosive wear*, in which cuffs, collars, turn-ups of trousers, bottom seams of dresses, pleat folds, buttonholes, and raised seams all show a particular kind of local breakdown. It is noteworthy that ordinary flat abrasion, which might be expected in garments at elbows, knees, and seats, is conspicuously absent. It has proved practicable to consider this group of damage types—erosive wear—as an entity whose likely onset can be predicted by methods described later in this paper.

2. BASIS OF ABRASION TESTING

In the course of our studies on the wear of textile materials it has been necessary to invent abrasion machines and to consider the methods of interpreting the data in practical terms. The general approach to this problem can be summarised under three headings—

(*a*) The abrasion machine should be capable of specification in engineering terms, so that a given fabric may be tested to give a reproducible numerical machine parameter. This criterion rules out the use of undefined abrading surfaces and actions by which machine results of only comparative use are derived. The abradant should not change with time or with use, and should be capable of precise reproduction in the workshop. We believe that this rules out the use of such abradants as emery, woven fabrics, and various undefined metallic abradants.

The actual abrasion machine should be heavily and soundly built, so that the action of the machine is unimpaired for a long period of use, up to, say, 10^9 cycles. The machine, to be of use as a routine instrument, should run at a reasonably high speed without impairing the action; this has the desirable consequence that the test conditions for most fabrics can be mild, giving a relatively high number of cycles to end-point in a satisfactory method.

(*b*) The end-point should be determined automatically by the machine. In many cases, particularly in flat abrasion, the end-point is determined visually by a subjective assessment. It is our experience that this leads to a much wider spread of individual results than is obtained with an automatic end-point, determined, say, by the breaking of a fabric. A satisfactory machine should therefore have an automatic end-point.

(*c*) All refinements of abrasion-machine design are in vain unless the results are related to the behaviour of the fabric in the field. We have therefore drawn freely on the results of field trials, and to these have added a study of fabrics whose commercial record is such that customer complaints on wear are virtually non-existent. It is remarkable how seldom the results of wear-testing machines have been checked against the field behaviour of the fabric tested. We may cite, as a brilliant exception to this, the article by Stoll[5], in which a number of fabrics were studied with success, judged by a remarkable prediction of the (U.S.) Quartermaster Combat-Course ranking from the laboratory results. The fact that, in the case of these Quartermaster fabrics, it was possible to set up a figure of merit, derived from several laboratory tests (comprising mechanical as well as abrasion tests), has been taken as one of the bases of our approach.

3. CHOICE OF TESTING MACHINES

Peirce[6] discussed the fundamentals of testing fabrics to foretell serviceability. In the case of resistance to wear, he recommended that, in general, imitative tests should be rejected. He recommended in their place tests of character, with simple conditions idealising the essential types of destructive action, not complex combinations of arbitrary features trying to imitate the accidental circumstances of practical use. Results of such testing should not be accepted as measures of serviceability but as complex characters to be correlated empirically with structure, etc. on the one hand, and with experience of service on the other. Further, the simplest tests should be used to their limit, the more complex tests being called in only when the simple ones prove insufficient.

We have accepted this appreciation of wear serviceability testing as the basis of our work. Peirce further made the point that a well made fabric is like a knot that may be teased apart with gentle fingers, but may be tightened by the application of brute force. We have therefore tried, as far as possible, to make our destructive tests gentle, well within the capacity of the fabric to take the stresses applied, so that cycle numbers have tended to be large. Our investigations have been, in the main, confined to woven fabrics in which the individual filaments of the yarn have been more or less free to move; i.e. they have not been embedded in a matrix such as oiled silk. Further, for the purpose of this paper, we propose to deal only with rayon staple fabrics which have been given a resin treatment of the crease-resisting type.

Machines proposed and available for the abrasive testing of textiles are now so numerous[7] that a choice based on systematic experimental comparison is no longer possible. However, all the devices so far proposed are eliminated by the first two criteria of the preceding section, and nearly all by the third criterion. In our studies on abrasion testing we have confined our attention largely to two machines—the BFT machine and the Wyzenbeek precision wear-tester.

The BFT machine was designed in the Bocking Laboratory, and it is proposed to publish a full description of it elsewhere shortly. For present purposes it will suffice to describe its essential features; a perspective view of the machine is given in Fig. 1. It consists essentially of a heavy platform, to the right of which is a steel runway on which a solid aluminium carriage *G*, mounted on ball races, reciprocates with a 0·5-in. stroke at 700 r.p.m. This motion is produced by a connecting rod *J* running to a crank-arm at the left of the platform; the crank spindle is geared to a six-decade counter *A*. The aluminium carriage and the head are furnished with rows of stenter pins on which the fabric (always a strip frayed down to 1 in. wide) is mounted. Above the carriage is the head *F*, which is capable of moving vertically in a heavy parallel link-motion *B*, counterpoised so

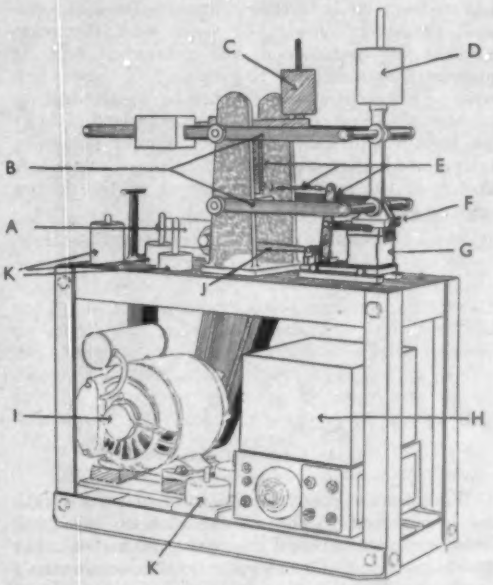

FIG. 1—BFT Mark III Testing Machine

that, with no weights on the head spigot, the whole of the head motion is in equilibrium; by placing weights on the spigot, definite vertical head loadings may be given. In the case of the flexing motion, which is adapted from the Stoll–Quartermaster machine, the flexing occurs round the end of a formed Staybrite FMB plate about 0·037 in. thick. The flexing plate is pulled to the left by a stirrup and link-motion *E* dead-weighted by a chain attached to a counterpoised bell-crank furnished with a weighting spigot. The essential improvement of the BFT over the Stoll machine is that the chain is attached to a dead point of the link-motion, so that the machine may run at a high rate with the weight *C* more or less motionless, thus removing large inertia effects. The design of carriage and head, with high thermal capacity and conductivity, is such that temperature rise during flex (or ball) abrasion is negligibly small.

The essence of the BFT flexing motion is the flex-plate. The edge of the stainless-steel plate is machined to a symmetrical 10° wedge, on the end of which a smooth section of a cylinder 0·017 in. in diameter is formed. It has been found extremely difficult to reproduce this edge, but we are now able to reproduce these flex-plates at will with a calibration value within 5% of the standard; it is a very advantageous form, insofar as any slight deviation from it is gradually reduced by abrasive wear in the course of flexing fabrics, so that a slightly malformed edge will become true on, say, running for 10 million cycles with a nylon fabric. In other words, the only effect that wear

has on the edge is to reduce imperfections of form and, extremely slowly, to wear back the edge so that the diameter of the cylindrical edge is increased, thus tending to give slightly slower flex wear. This point can be checked by regular testing of the abrading edge with a standard fabric: we have found that the plate should generally be replaced after about 10^6 machine cycles. A sketch of the essential dimensions of the flexing edge is given in Fig. 2.

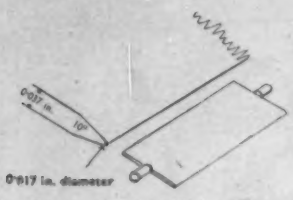

FIG. 2— Details of Flexing Plate

When the machine is in use, the 1-in. strip of fabric is attached to the stenter pins on the fixed head, and passed round the flexing plate, the other end being attached to the pins on the reciprocating carriage. The head and the flex-tension spigot are each weighted and the machine is set in motion. At the end of the test the fabric breaks, and the flexing plate moves forward under its loading, so that the loading weight falls and knocks off the motor by means of the micro-switch. The number of revolutions to break is indicated on the counter.

The BFT machine has a further mechanism, which may be termed the *ball-penetration test;* here a $\frac{1}{16}$-in. ball-bearing is fitted to an insulated collet attached to the fixed head of the machine. The fabric strip for this test is mounted across the top of the platform of the reciprocating carriage, the ball is loaded by a dead-weight on the head spigot, and the machine is set in motion and taken to an end-point given by penetration of the fabric and electrical contact of the ball with the platform of the reciprocating carriage. In our first experiments we used a 3-ma. relay to knock the machine off at the instant of contact. This current was sufficient to cause scarring of the ball-bearing by arcing, which could reduce the indicated cycles by a factor of 2–4. In the present Mark III of the BFT machine, the electrical mechanism used for stopping the motor on ball penetration is a thyratron relay *H* (Fig. 1) working at about 2 μa., a current too small to produce observable scarring of the ball surface. After every few tests the ball is moved to give a new point of contact, and a new ball is inserted daily. We have found that commercial ball-bearings give extremely reproducible results.

The BFT machine conforms to the engineering requirements set out above, insofar as the end-point is automatic and not subjective, and the abrading elements are reproducible and of satisfactory life. The instrument gives extremely good reproducibility, so much so that we are now certain that the variations observed are usually due to real variations in the fabric examined. We have investigated the statistics of the motions in detail: on flexing a reasonably uniform fabric, the mean of six results is reliable within $\pm$ 10% at a 95% level of probability; the statistics of the ball-penetration test are similar.

In choosing a flat abrasion machine, the claims of a number were examined, and of those available we chose the Wyzenbeek precision tester as being most useful for our purposes. This is essentially a flat abrader; the abradant is mounted on a reciprocating cylinder, and the fabric pressed on to it by means of a dead-weight. The end-point in routine testing is judged subjectively by the production of a hole or a threadbare appearance. As abradant we chose a wire gauze rather than a woven textile, since it was found that such a material was reproducible and stable. The actual gauze used was a 28-gauge wire, woven 20 meshes to the inch. This, after preliminary polishing, has proved to be a very satisfactory abradant. Reproducibility, presumably because of difficulties with the subjective end-point, is not as good as with the BFT machine, but the mean of six results is reliable to $\pm$ 25% at a 95% level of probability.

Testing conditions were standardised early and, in this paper, all results quoted were produced with the following standard conditions—

PLATE FLEXING— 4 lb. head weight and 4 lb. tension on the flexing plate for a 1 in. wide strip of fabric.

BALL-PENETRATION TEST — $1\frac{1}{2}$ lb. head weight.

WYZENBEEK TESTER— Pressure $6\frac{1}{2}$ lb. applied on a 2 in. square pad to a $2\frac{1}{2}$ in. wide strip of fabric tensioned at 4 lb. The stroke of the Wyzenbeek machine is $4\frac{3}{4}$ in.

4. MACHINE FACTORS

The BFT and the Wyzenbeek machines, used as indicated, give three contrasted abrasion tests. A number of other motions have been investigated, and have been abandoned either for reasons of sound mechanics or for the technical reason that they were compounded largely of one or more of the other motions. The three motions used for mechanically destroying woven fabrics are quite different in their essential mechanisms; it is not yet certain that the mechanical breakdown of a given fabric can be characterised by these three motions alone, but we have reached the practical conclusion that no radically differing motions have been proposed in the literature nor have we been able to find any. We have therefore adopted a tentative working hypothesis that the mechanical breakdown of a woven fabric can be characterised by a limited number of mechanisms, which for the present we equate to flat abrasion, a flexing test, and a ball-penetration test.

For practical purposes the results of machine tests should be normalised to give numbers of reasonable size and comparable range. The normalisation factors applied are—

Ball-penetration test	10^{-2}
Flex test	10^{-3}
Flat abrasion test	10^{-3}

The normalised machine factors we denote in this paper by B, P, and F to represent results of the ball-penetration, flex, and flat abrasion tests respectively. It has not yet been possible to relate the values of B, P, and F as determined on a given fabric to a combination of ultimate fibre properties and fabric structure factors, but we have no doubt that this problem is susceptible of rational analysis along lines suggested by Hamburger [8], who related the flat abrasion of single yarns to their mechanical properties. Our experience of handling these factors, however, has given us some idea of the complex qualities they represent.

The flex test value P is related to the behaviour of the fabric in internal motion, and, in effect, this test is perhaps the application of the "nimble fingers" of Peirce, mentioned above. It is related certainly to the coherence of the fabric structure and also, in general, to its ruggedness, which is presumably a function of the ruggedness of the individual fibres. Microscopic examination of a fabric slowly flexed round a plate shows that the structure is in considerable internal motion; that, not only do the yarns bend as they go round the flex edge, but also they twist and untwist in quite remarkable fashion. The debris from the flex test suggests that the fibres fail by tensile breaks; the internal motion of the structure tends to set up local stresses, which may occasionally be so high that an individual fibre or filament is broken. This view tends to be confirmed by the observation that the P value is highly dependent upon the lubricity of the structure. The addition of fibre lubricants increases P, and the anti-lubricating effect of a finish which binds fibres or filaments together tends to diminish P. The

TABLE I

Effect of Lubrication on Flex Results

(Fabric—Utility 1005)

State	P Value Wet	P Value Dry
(1) Commercially scoured...	1·5	15
(2) Laboratory-scoured	—	7
(3) As (2), soap not completely removed	—	41
(4) As (2), extracted with benzene–methanol	1·0	2·3
(5) As (2), softened with 0·1% Gemex Z6	1·0	22
(6) Tested wet with olive oil	—	59

enormous effect of lubrication is shown by the data of Table I, which is self-explanatory. On the whole, the effects of flexing may be described as the application of short-range elastic strains to the interior of the fabric; these are of an oscillatory nature, so that they tend to break down coherence, and this breakdown is accelerated by lack of lubrication.

The ball-penetration test value B depends to some extent on fabric structure factors, but it is most nearly related to the toughness or lack of brittleness of the individual fibres: a treatment which increases fibre brittleness reduces the B value considerably. Lubrication has a comparatively small effect upon B, increase of lubrication tending to decrease the B value a little.

The flat abrasion value F is related to the attrition of the surface fibres from a fabric in which internal motions are either absent or are much less important than in the flex test. It is largely independent of the abrading surface. In a very extended series of F values, ranging from a tendered viscose rayon fabric to a well constructed nylon fabric, it was found, in general, that the same ranking is given with mild textile abradants, with wire gauze, and, if certain precautions are observed, even with the quick abrasion of carborundum. This is a general statement only; for in other short-range series, reversals of ranking are found with different flat abradants. It should be noted that most practical flat abradants do not have a uniform flat surface and present sufficient projections to cause local flexing of the fabric. For the present, we attribute small differences in ranking between different flat abradants to this component of flexing.

The three machine factors are normally determined under the usual laboratory conditions of 65% R.H. and 20°C. We have found, in studies not reported in this paper, that in some instances it is important that they be determined at other humidities in addition—say at 30, 45, 80, 90% R.H. and wet—in order to get reasonable correlation with field wear.

5. INDEPENDENCE OF MACHINE FACTORS

An important conclusion is that, in a given fabric, the machine factors P, B, and F, being generally related as to level, will have, at that level, considerable independence. This may be made clearer by noting that a given fabric, on chemical tendering, will show losses in all three factors, but the proportion of loss will not be the same for each. However, in a range of fabrics of varying structure and finish but of approximately constant weight and character, the relative importance of the factors is found to vary considerably. This may best be demonstrated by defining three new quantities—

$$\overline{P} = \frac{P}{P + B + F}$$

$$\overline{B} = \frac{B}{P + B + F}$$

$$\overline{F} = \frac{F}{P + B + F}$$

so that $$\overline{P} + \overline{B} + \overline{F} = 1$$

In Table II are given the machine factors (at 65% R.H. and 20°C.) and their derivatives for (*a*) a range of dress-weight materials and (*b*) a single fabric (Utility quality U 1009) given different commercial finishes. It will be noted that there is considerable variation, not only in general level but also in the proportional contribution of each of the machine factors. Since the sum of $\overline{P}$, $\overline{B}$, and $\overline{F}$ is unity, we are able to plot the relative proportion of each of these quantities on a triangular diagram. The derived quantities given in Table II are set out in Fig. 3 and 4 for the dress-weight and the U 1009 range respectively.

TABLE II

Machine Abrasion Results on a Series of Dress-weight Rayon Staple Fabrics

Component Yarns*	Resin Content (%)	B	P	F	D	$\bar{B}$	$\bar{P}$	$\bar{F}$
Fa: Sv; v–a	9·1	1·2	2·8	1·0	1·7	0·24	0·56	0·20
Fa: Sv–a	6·4	1·2	1·6	2·0	1·4	0·25	0·33	0·42
Fa: Sv–a	5·7	0·8	1·6	1·0	1·1	0·24	0·47	0·29
Fa: Sv–a	5·4	5·5	1·1	0·5	1·8	0·78	0·16	0·06
Fv; a: Sv–a	9·3	0·6	1·6	2·5	0·9	0·13	0·34	0·53
Fa: Sv–a	6·5	0·9	2·3	17·0	1·3	0·05	0·11	0·84
Fa: Sv–a	7·7	0·7	2·0	1·0	1·0	0·19	0·54	0·27
Fa: Sv–a	4·0	1·1	4·0	2·0	1·7	0·15	0·56	0·29
Fv; a: Sv–a	7·1	1·2	2·6	4·5	1·6	0·14	0·31	0·55
Fa: Sv–a	9·2	0·8	2·5	2·0	1·2	0·15	0·47	0·38
Fa: Sv–a	Nil	1·3	1·3	9·0	1·5	0·11	0·15	0·74
Fa: Sv–a	5·0	1·1	3·1	1·5	1·6	0·19	0·54	0·27
Sv	11·8	4·7	5·6	3·0	5·1	0·35	0·42	0·23
Sv	11·9	4·4	11·3	0·75	6·3	0·27	0·69	0·04
Sv–a	12·3	1·6	5·3	1·0	2·5	0·20	0·67	0·13
Fa: Sv	6·8	1·0	4·4	1·0	1·6	0·16	0·68	0·16
Fa: Sv; v–a	8·7	0·9	3·6	1·0	1·4	0·16	0·65	0·19
Fa: Sv–a	7·7	0·8	4·0	3·0	1·3	0·10	0·51	0·39
Fa: Sv–a	8·2	1·5	2·2	1·0	1·6	0·32	0·47	0·21
Fa: Sv–a	10·7	2·1	1·7	2·0	1·3	0·36	0·29	0·35
Sv	13·5	3·5	6·8	2·0	4·6	0·28	0·55	0·17
Sv	11·8	2·1	6·4	1·5	3·1	0·21	0·64	0·15
Sv–a	0·55	3·5	0·5	1·5	0·9	0·64	0·09	0·27
Fv; a: Sv–a	9·8	0·8	1·6	7·0	1·1	0·09	0·17	0·74
Fa: Sv	7·6	2·4	6·3	1·5	3·4	0·24	0·62	0·14
Fa: Sv–a	6·7	1·7	4·4	1·5	2·4	0·22	0·58	0·20
Fa: Sv–a	9·1	1·7	1·3	1·0	1·5	0·43	0·32	0·25
Fa: Sv–a	3·8	0·9	5·1	7·5	1·5	0·07	0·38	0·55
Fa; v: Sv–a	9·6	1·0	1·0	1·5	1·0	0·20	0·29	0·42
Fa; v: Sv–a	5·0	1·1	3·1	2·0	1·9	0·12	0·66	0·22
Fa: Sv–a	8·0	2·6	0·6	17·0	1·0	0·13	0·03	0·84
Fv; a: Sv–a	6·9	2·0	2·0	1·5	2·0	0·36	0·36	0·18
Sv	12·8	2·5	4·2	4·0	3·0	0·23	0·39	0·38
Fa: Sv–a	Nil	9·9	2·6	1·0	4·1	0·73	0·19	0·08
Fv; a: Sv–a	Nil	1·1	3·4	1·0	3·8	0·48	0·40	0·12
Fa: Sv	Nil	5·2	10·3	2·0	6·9	0·30	0·59	0·11
Fv; a: Sv–a	Nil	7·1	5·6	2·0	6·2	0·48	0·38	0·14
Fa: Sv–a	Nil	1·5	6·7	7·5	2·4	0·10	0·43	0·47
Fa: Sv–a	Nil	0·8	3·5	2·0	1·3	0·13	0·56	0·31
Fv: Sv	Nil	5·4	11·1	4·0	7·2	0·26	0·54	0·20
Sv–a	6·0	1·5	2·3	3·0	1·8	0·22	0·34	0·44
Sv–a	Nil	3·8	5·5	4·0	4·5	0·29	0·41	0·30
Sv	7·8	33·7	14·7	4·0	20	0·64	0·28	0·08
Sv	Nil	15·3	29·5	14·0	20	0·26	0·59	0·24
Sv	10·3	1·8	1·6	20·0	1·7	0·08	0·07	0·85
Sv	Nil	5·8	30·3	16·0	9·7	0·11	0·58	0·31
Sv	11·9	4·8	2·3	6·0	3·1	0·37	0·18	0·45
Sv	Nil	7·6	18·3	9·0	11	0·22	0·52	0·26
Sv	7·3	6·1	26·0	6·0	9·9	0·16	0·68	0·16
Sv	Nil	3·9	24·8	10·0	6·7	0·10	0·64	0·26
Fv; a: Sv–a	Nil	2·1	2·9	1·0	2·4	0·35	0·48	0·17
Sv	Nil	8·4	22·1	2·0	12	0·26	0·68	0·06
Fa: Sv	Nil	18·2	6·1	3·0	9·1	0·67	0·22	0·11
Sv	Nil	45·6	8·7	14·0	12	0·67	0·13	0·20
Fv; a: Sv–a	Nil	5·6	1·5	1·5	2·4	0·66	0·17	0·17
Fa: Sv–a	0·7	1·3	0·8	3·5	1·0	0·23	0·14	0·63
Fv: Sv	7·4	0·8	5·8	2·5	1·4	0·09	0·64	0·27
Fa: Sv; v–a	Nil	3·5	2·0	3·0	2·5	0·41	0·24	0·35
Fv: Sv	Nil	7·7	6·9	19·0	7·3	0·23	0·21	0·56
Fa: Sv–a	Nil	4·5	1·3	5·0	2·0	0·42	0·12	0·46
Fv: Sv	Nil	5·0	0·7	0·5	5·7	0·27	0·37	0·36
Sv–a	Nil	0·9	1·5	13·0	1·1	0·06	0·10	0·84
Fa: Sv	Nil	0·7	2·1	13·0	1·1	0·04	0·13	0·83
Sv–a	Nil	15·0	15·5	18·0	15	0·31	0·32	0·37
Fa: Sv	Nil	9·1	8·9	4·5	9·0	0·40	0·40	0·20
U 1000 Sv	12·5	5·8	32·2	4·0	9·8	0·14	0·77	0·09
	12·0	5·4	46·5	14·0	9·7	0·08	0·71	0·21
	12·0	1·9	149·0	20·0	3·6	0·01	0·87	0·12
	Nil	10·3	29·5	4·0	15	0·26	0·67	0·07
	Nil	8·4	40·6	4·0	14	0·16	0·77	0·07
	10·2	4·4	45·5	3·0	8·0	0·08	0·86	0·06
	14·2	4·2	41·4	4·0	7·6	0·08	0·84	0·08
	10·7	1·6	23·8	5·5	3·0	0·05	0·77	0·18
	12·7	5·5	68·4	12·0	10	0·07	0·78	0·15
	5·1	0·5	31·7	2·5	14	0·22	0·73	0·05
	3·5	8·8	70·1	26·0	16	0·08	0·67	0·25
	5·7	12·0	48·6	3·0	19	0·19	0·76	0·05
	10·9	0·9	5·1	10·0	1·5	0·04	0·20	0·76

TABLE II (continued)

Component Yarns*	Resin Content (%)	B	P	F	D	$\bar{B}$	$\bar{P}$	$\bar{F}$
U 1000 Sv (continued)	11·1	0·9	3·2	6·0	1·4	0·09	0·32	0·59
	8·0	2·0	47·5	10·0	3·8	0·04	0·80	0·16
	8·6	1·6	3·7	1·5	2·2	0·24	0·54	0·22
	11·6	1·0	31·7	2·0	1·9	0·03	0·91	0·06
	14·2	1·9	180·0	3·0	3·8	0·01	0·97	0·02
	13·8	1·6	149·4	3·7	3·2	0·01	0·97	0·02
	14·7	1·0	20·5	0·6	1·9	0·05	0·93	0·02
	16·4	1·5	19·4	6·0	2·8	0·06	0·72	0·22
	13·9	3·8	19·8	4·5	6·4	0·14	0·70	0·16
	8·3	2·0	25·9	4·5	3·7	0·06	0·80	0·14
	12·2	4·3	20·7	2·0	7·1	0·16	0·77	0·07
	10·9	8·5	32·7	16·0	14	0·15	0·57	0·28
	12·8	3·6	9·1	2·5	5·2	0·24	0·60	0·16
	11·6	0·9	116·6	1·2	1·8	0·01	0·98	0·01
	10·4	0·6	62·7	5·0	1·2	0·01	0·93	0·06

* Fabric construction is coded—

F = Filament a = acetate rayon
S = Staple fibre v = viscose rayon
v–a = Blend of viscose and acetate rayon staples

(:) A colon separates staple and filament components

(;) A semi-colon shows the use of more than one type of staple or filament

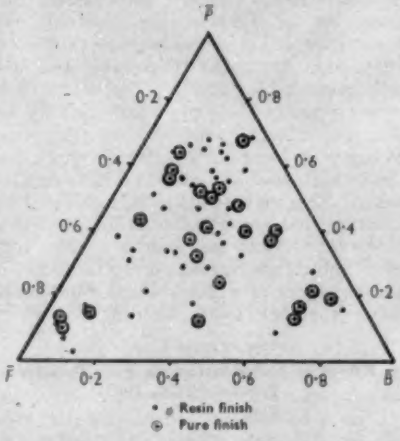

FIG. 3— Relative Values of Ball, Flex, and Flat Abrasion Results for a Series of Dress-weight Rayon Staple Fabrics

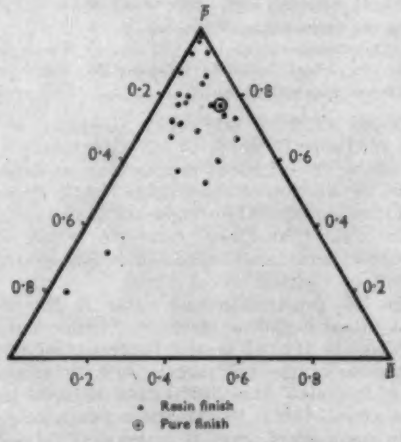

FIG. 4— Relative Values of Ball, Flex, and Flat Abrasion Results for a Series of Commercial Finishes on Quality U 1000

The sixty-five fabrics surveyed in Fig. 3 cover virtually the whole area of the triangular diagram, thus indicating that in the range of fabrics studied the three factors are found combined in practically all conceivable proportions; this conclusion will obviously remain valid if the testing is extended from dress-weight fabrics containing viscose and acetate rayons to any further range of fabrics of differing type and fibre composition. In Fig. 3 the pure-finish fabrics and resin-finish fabrics are separately indicated, and it appears that the general conclusion is true for both sets of fabrics. Fig. 4 records the behaviour of a single rayon staple quality (U 1009) as produced by a series of commercial finishes; the fabric, both in pure finish and in a variety of crease-resistant finishes, tends to have a relatively high resistance to flex abrasion and to be relatively susceptible to ball penetration and flat abrasion.

It is clear, therefore, that the three machine factors, P, B, and F, are independent quantities characterising the mechanical breakdown of a fabric. It is thus possible to have a fabric which is relatively resistant to flex abrasion, ball penetration, or flat abrasion, or to any two of these quantities. It is certainly not possible to deduce the level of, say, flex and ball test results from a knowledge of the results of the flat abrasion test, to which wear testing is usually confined.

Following on these considerations, we have adopted a second working hypothesis, that it is possible to foretell wear serviceability by a figure of merit derived mathematically from the machine factors possibly combined with other factors, e.g. tear strength and bending length.

6. THE CONCEPT OF DUTY FACTOR

In the course of our testing of crease-resist-finished heavy-duty rayon staple fabrics we noted that the flat abrasion F gave no guide, and might indeed be very high in a fabric known to be unserviceable. After due consideration, we ignored this factor in our primary treatment. It was further observed that every known failure had either a low B value or a low P value, or both, whereas satisfactory fabrics had relatively high B values and P values. This was shown clearly by setting out the P values along the x axis and the B values along the y axis and then joining the points so marked by a line as in Fig. 5. Typical data for fabrics known to fail are the lines AA and $A'A'$; corresponding lines for satisfactory fabrics are MM and $M'M'$. In our first treatment, we drew the line OD bisecting the angle between the x and y axes, and found that the intercept made by the performance line on OD was, at first view, an indication of performance. Other lines, such as OD' and OD'', could be drawn, on which the intercepts were weighted towards the P values and the B values respectively. An elementary analysis of this diagram shows that the intercepts along OD are proportional to the harmonic mean of B and P, and those along OD' and OD'' to weighted harmonic means of the same quantities.

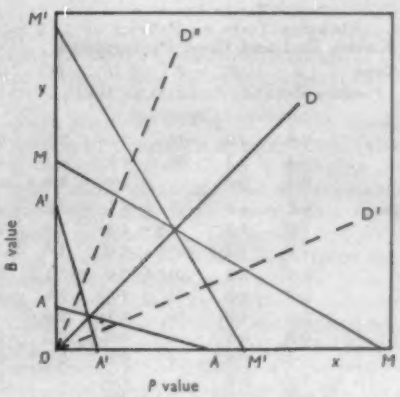

FIG. 5—Concept of Duty Factor

In general, the weighted harmonic mean H' of a series of quantities $R, S, T, \ldots$ is defined by the expression—

$$\frac{1}{H'} = \frac{\alpha}{R} + \frac{\beta}{S} + \frac{\gamma}{T} + \ldots$$

where $\alpha, \beta, \gamma, \ldots$ are the numerical constants denoting the relative weighting of $R, S, T, \ldots$ respectively, chosen so that—

$$\alpha + \beta + \gamma + \ldots = 1$$

In the unweighted harmonic mean—

$$\alpha = \beta = \gamma = \ldots = \frac{1}{n}$$

where n is the number of terms $R, S, T, \ldots$

For the two quantities P and B, the unweighted harmonic mean D therefore becomes—

$$D = \frac{2BP}{P+B}$$

The harmonic mean may be weighted towards P or towards B; for a twofold weighting, the new means become—

$$D' = \frac{3BP}{2B+P} \quad (P \text{ overweighted})$$

and

$$D'' = \frac{3BP}{B+2P} \quad (B \text{ overweighted})$$

The harmonic mean of P and B is the *duty factor* D of the fabric examined. The duty factor is determined normally by flexing a warp strip with the face of the fabric towards the flexing edge and combining this with a ball-penetration test with the face of the fabric upwards and the ball reciprocating in the weft direction; this is termed the *warp duty factor*. In the same way, a *weft duty factor* can be obtained, and an arithmetic mean of the two can be taken as the duty factor of the fabric. In practice, for most commercially useful heavy-duty fabrics, the difference between warp and weft can be ignored, and a warp duty factor alone determined.

In Table III are set out the testing data for a range of unsatisfactory fabrics and for a similar range of satisfactory fabrics. The weighted harmonic means D' and D'' are recorded in addition to the duty factor D. This table gives every result

TABLE III
Abrasion Tests on Fabrics of Known Bad and Good Performance

Type	*B*	*P*	*F*	*D*	*D'*	*D''*
FABRICS SHOWING PREMATURE WEAR						
Commercial Complaints						
Slacks	2·6	2·5	26	2·6	2·5	2·6
	9·4	3·2	58	4·8	4·1	5·8
	2·2	5·0	—	3·0	3·6	2·8
Gym slips	6·2	4·8	9	5·4	5·2	5·7
	3·0	1·3	0·8	1·8	1·6	2·1
	7·8	1·1	—	2·0	1·5	2·6
Overalls	2·6	3·5	8	3·0	3·1	2·8
	3·6	4·0	7	3·8	3·9	3·7
	2·6	1·8	10	2·1	2·0	2·3
	0·6	0·5	1	0·6	0·5	0·6
	0·8	0·8	1	0·8	0·8	0·8
	2·6	0·8	—	1·2	1·0	1·4
	1·4	7·5	0·1	2·3	3·1	1·9
Sports shirts	1·1	9·2	0·8	2·0	2·7	1·6
	1·6	10·1	0·8	2·8	3·6	2·2
	2·6	12·6	15	4·4	5·5	3·5
	1·5	6·9	—	2·4	3·1	2·0
Work skirts	2·1	2·0	—	2·1	2·0	2·1
Costumes	11·3	1·1	10	2·0	1·6	2·8
Kilts*	1·0 to 3·8	0·2 to 1·0	—	0·3 to 1·6	0·3 to 1·3	0·4 to 3·8
Unsatisfactory Field-trials						
Sports shirts	1·1	12·6	—	2·2	2·8	1·6
Gym slips	3·1	5·3	1·5	4·0	4·3	3·6
Overalls	2·4	2·5	10+	2·5	2·5	2·4
	0·6	5·1	4	1·1	1·7	1·0
	1·7	131·5	10+	3·4	5·0	2·5
FABRICS OF SATISFACTORY WEAR						
Satisfactory Field-trials						
Slacks	8·8	18·4	11	12	14	11
	14·8	5·7	20	8·2	7·2	9·7
	5·3	33·6	10	9·2	12	7·4
	16·7	4·5	—	7·0	5·9	8·8
Gym slip	18·6	26·6	9	22	23	21
Overall	24·7	46·4	40	32	36	29
Shirts	3·2	300	—	6·4	9·4	4·8
	22·5	600+	—	44	63	33
	23·1	250	—	42	58	33
	7·1	52·3	11	12	17	10
	5·2	150+	11	10	15	7·7
Raincoats	160	63·5	—	91	80	111
	12	35·3	—	18	22	15
	5·6	52·4	—	10	14	8·0
Suiting (blazer)	8·9	7·1	4	7·9	7·3	8·2
Fabrics of Satisfactory Commercial Reputation						
English slacks	18	10	30	13	12	14
American slacks and suitings	30·8	91	—	46	56	40
	6·7	9·4	—	7·8	8·3	7·4
	5·1	15·1	—	7·6	9·1	6·5
	26·9	160	—	46	60	33
Sports shirts	13·2	73·5	—	22	29	18
	6·8	39·8	—	12	15	9·5
Canadian slacks	37·5	48·1	20	42	44	41
	8·1	14·2	6	10	11	9·4
	6·8	41	30	12	15	9·4
	14·4	13·6	5	14	14	14
Light suitings	21·7	19·4	45	21	20	21
	20·5	48·7	70	29	33	25
	20·2	3·5	38	6·0	4·8	7·8
	17·1	19·2	6	18	18	18

*Nineteen fabrics

available in the Bocking Laboratory up to December 1954 for heavy-duty fabrics which have been the subject of justified commercial complaint of erosive wear, and all cases of failure of heavy-duty fabrics by premature erosive wear in field trials. The satisfactory fabrics comprise all cases available where field trials have shown the heavy-duty fabric to be entirely satisfactory in wear and free from erosive wear effects. To these is added a range of British, American, and Canadian fabrics whose commercial record is such that we may presume that poor wear is never encountered. There are about thirty fabrics in the failure group and a similar number in the satisfactory group. It should be noted that there is no overlap of *D*; we therefore regard *D* as usefully diagnostic of the possibility of erosive wear occurring in the normal use of a crease-resisted heavy-duty rayon staple fabric.

The duty factor is an instance of a figure of merit of the simplest type, being derived from two factors only. The method of combination as a harmonic mean was achieved empirically as noted above. Of the means of a set of values, the harmonic mean is smaller than the geometric mean, which is itself smaller than the arithmetic mean: the harmonic reflects the low values of the set. If erosive wear is dependent on two components related to the ball-penetration and flex machine factors, then a low duty factor tends to show better than the other simple means a deficiency of either component, since *D* can never have a value greater than twice the lower of either the *P* value or the *B* value. Other mathematical treatments exist which will emphasise even more sharply than the harmonic mean deficiencies in any series of machine factors, but an empirical examination of such treatments indicates that they are not superior to the harmonic mean in the present case.

7. PREDICTION OF WEARABILITY BY MEANS OF THE DUTY FACTOR

The data of Table III are comprehensive: that is to say, the table contains the test data on every fabric available to us which is known to have failed by reason of erosive wear, and, on the other hand, on all the fabrics tested which are of known satisfactory behaviour or commercial reputation. There has been no elimination of any anomalous results to make the table more homogeneous. In the case of the duty factor *D* there is no overlap between the failures and the good fabrics. When the harmonic mean is biased slightly towards the flex results or the ball-penetration results, as in *D'* and *D''* respectively, correlation between wear and derived factor is not so good, since in both cases there is a little overlap. Any further degree of biasing will clearly, from an inspection of the *B* and *P* values in the table, lead to a greater degree of overlap.

We are therefore satisfied that the duty factor *D*, the unweighted harmonic mean of *B* and *P*, is the most valuable diagnostic factor, for prediction of erosive wear in resin-treated rayon fabrics, which can be derived from the data of Table III. Furthermore, we have noted that when the fabrics

which are the subject of complaint have very low duty factors, they are those which have broken down in the shortest periods. We have adopted a third working hypothesis: the magnitude of the duty factor of a resin-treated rayon staple fabric is strongly correlated with its freedom from premature erosive wear.

The duty factor thus represents a laboratory test which is superior to any hitherto used in the assessment of the wear behaviour of resin-finished spun rayons. Certain technical aspects of the duty factor must be emphasised—

(*a*) The duty factor is intended to be used only with pure and blended rayon staple fabrics to which the normal types of crease-resistant finishes have been applied. The test is based on two machine factors determined at one relative humidity; it seems likely that the success of the duty factor as a diagnostic figure depends on the fact that at relative humidities higher or lower than 65%, and, indeed, wet, the duty factors of this particular type of fabric are largely unchanged. *D* is not a guide to the wear properties of spun rayons in pure finish; the wear there is of a different type from the erosive wear discussed in this paper. It is not certain that the duty factor in its present form and at its present levels is suitable for application to cotton and resin-treated cotton fabrics.

(*b*) Determination of duty factor is simple and can be used as a routine check. The BFT machine is rapid in action; it has high reproducibility, so that observed variations are almost always due to real variation in a fabric from place to place. Duty factor, too, is distinguished from most other abrasive tests insofar as the scale is very open: in the present series there is a difference of a hundredfold between the worst and the best fabrics.

(*c*) The duty factor of the treated fabric can, with a good treatment, be as high as, or higher than, the harmonic mean of *B* and *P* determined on the untreated fabric; i.e. by this method of assessment the apparent resistance of the fabric to abrasion at 65% R.H. can be improved by application of a resin finish.

(*d*) The duty factor predicts erosive wear, i.e. at seams, collars, turn-ups, button-holes, etc. It does not necessarily predict resistance to flat abrasion. In testing a new fabric, therefore, a flat abrasion test should be carried out, but, from our experience in field trials, the minimum level of flat abrasion can be set much lower than the level usually adopted as diagnostic of poor wear in resin-treated fabrics. This is based on the observation that very few fabrics, even those showing bad erosive wear, do not tend to wear at elbows, knees, etc. by a strict flat abrasion mechanism. In practice, with a fabric of known reliable construction, it is usually permissible to omit a flat abrasion figure provided that the duty factor is good. Should a flat abrasion test be required, we recommend for spun rayons in heavy-duty wear a level of 2,000–3,000 on the Wyzenbeek machine under the conditions specified previously. In the same way, it is usual also to test resin-finished spun rayons by a tear test. Here, too, it is found that, with a fabric of known good construction, it is not necessary to carry out this test if the duty factor is reasonable, unless the finish is abnormally stiff.

(*e*) In the light of our experience, based upon the testing of thousands of commercial fabrics, we have ventured to set down a tentative scale of duty factors for crease-resisted rayon staple fabrics designed for different end-uses. The suggestions are shown in Table IV. The levels given for "satisfactory" wear are such that a garment will have a reasonable wear-life and will not give rise to consumer complaint. It must be emphasised here that the levels are given for specific end-uses, and the employment of a satisfactory dress fabric in an overall—a not infrequent occurrence—may give unsatisfactory wear unless the fabric is chosen to comply with the level for overalls.

TABLE IV

Levels of Practical Duty Factor on Rayon Staple Fabrics with a Crease-resistant Finish

Fabric Type	Satisfactory Wear	Outstanding Wear	Wear Properties > Practical Requirements
	LIGHT-DUTY GROUP		
Dress, etc. fabrics	1–4	4–7	7+
	HEAVY-DUTY GROUP		
Gym-slips, slacks, men's suitings	6–10	10–20	20+
Shirtings	5–8	8–15	15+
Overalls	7–12	12–25	25+

Garments made from fabrics of "outstanding" wear levels may be expected to be discarded for reasons other than wear, especially those at the upper limit. There appears, therefore, to be no practical advantage in attempting the construction of fabrics at higher levels of wear properties than are given in the third column of the table.

8. COMPARISON OF BFT WITH OTHER WEAR-TESTING MACHINES

There is a general belief amongst those responsible for the testing of fabrics that the use of flat-abrasion machines to determine the suitability for wear of resin-treated rayon staple fabrics leads to uncorrelated results. We have been able to confirm this belief: the Wyzenbeek figures of Table III show quite clearly that in about one case in five the flat-abrasion test does not give warning that the fabric has poor wear-properties. The same is likely to be true for all machines which have essentially a flat-abrasion motion. We have examined the relation between duty factor determined on the BFT and the results obtained for the same set of fabrics on other machines.

In Fig. 6 we give a plot for U 1009 quality of duty factors against test results on the Linra abrasion tester. It will be seen that there is virtually no correlation between the two sets of test figures. We draw attention to the fact that a large proportion of the fabrics which would be rejected by the duty factor test are passed by the Linra machine; and, conversely, fabrics which would be eliminated

by the Linra contain about equal proportions of fabrics acceptable and unacceptable by the duty factor test.

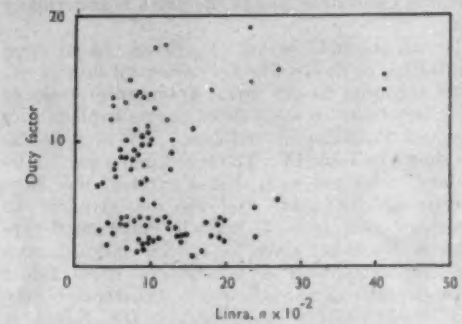

FIG. 6—Comparison of BFT and Linra Results on a Series of U 1009 Fabrics

Fig. 7 is a plot of duty factor against Martindale machine tests (with woven-wool abradant). All the tests were made on a single rayon staple gaberdine. The fabrics with a simple urea–formaldehyde finish are grouped together, whereas those containing a methacrylate addition are shown by the Martindale to be improved, although the duty factor suggests no improvement. The duty factor of the untreated fabric is also plotted. It may be said that there are three separate and distinct groups having separate correlations, depending on whether they are pure finish, simple resin finish, or resin plus methacrylate finish.

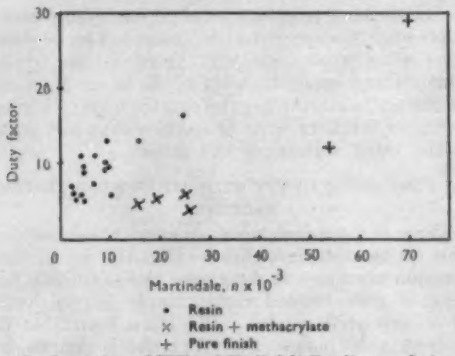

FIG. 7—Comparison of BFT and Martindale Results on a Series of Rayon Gaberdine Fabrics

The position can be made clearer if the statistics of a pass–fail criterion applied on two different instruments are considered. In Table II the machine-abrasion data for a series of finishes on U 1009 quality fabric are tabulated; we may reasonably set up two sets of criteria for the BFT and Wyzenbeek machines respectively—

$$\text{Good level} \quad \begin{cases} D < 3 \\ F < 3 \end{cases}$$

$$\text{Excellent level} \quad \begin{cases} D < 5 \\ F < 5 \end{cases}$$

The classification of the 27 fabrics given in Table V makes it clear that correlation between D and F is poor and that, at either level, there is an important group of fabrics for which the two machines give opposed conclusions; this indeterminate group is larger, relative to the group of fabrics tested, at the higher level.

TABLE V

Results of Testing U 1009 Fabrics on BFT and Wyzenbeek Machines

	No. of Fabrics at— Good Level	Excellent Level
Pass by both D and F ...	16	4
Pass by D; fail by F ...	3	9
Pass by F; fail by D ...	4	7
Fail by both D and F ...	4	7
Total No. of fabrics tested ...	27	27

Our general experience may thus be summed up: machines having essentially flat-abrasion motions — Linra, Martindale, Ring–Wear, Schiefer, Taber, Wyzenbeek, etc.— are poor guides to premature erosive wear on resin-finished rayon staple fabrics. The data presented here show that the duty factor, determined under the prescribed conditions, is much to be preferred in this respect.

9. CONCLUSIONS AND PROSPECT

We consider that the work detailed in this paper represents an advance in the art of abrasion testing. The BFT Mark III machine is a departure on rational engineering principles and provides a laboratory tool which is rapid, precise, and reproducible. The machine has been applied to the study of a technically important group of fabrics, viz. heavy-duty spun rayons with a crease-resist finish, in which, it is generally understood, a poor finish can result in poor wear. Using complaints, field-trials, and commercial experience with satisfactory fabrics, we have been able to define a duty factor computed from two different abrasion machine factors. This method of analysing abrasive wear into diverse machine factors capable of recombination into a figure of merit has proved fruitful in the present instance, and we believe that it can be fruitfully applied to other problems of correlation of field wear with laboratory testing.

The machine is a convenient laboratory tool. As well as its use, in the way previously discussed, as a method of checking fabrics before sale, it has other important prospective uses in the hands of the technologist. It can be applied—

(*a*) To investigate plant conditions precisely to find the combination of conditions most advantageous in the production of good-wearing fabrics.

(*b*) In laboratory investigations, to the development of improved finishes, without embrittlement and with adequate interfibre lubrication or at least interfibre freedom.

(*c*) As an aid to the fabric designer in producing rayon staple blended fabrics particularly suited to heavy-duty use. Its use here is illustrated by the data of Table VI, where the importance of composition of blend in a constant structure is well

brought out. We propose in the near future to use the duty factor method to assess the relative merits in wear of various fabric structures woven from the same yarn.

TABLE VI

Dependence of Duty Factor on Fibre Composition

Composition	D
85 : 15 Fibro–nylon ...	29
100% Fibro	16
67 : 33 Fibro–Fibrolane ...	12
75 : 25 Fibro–Fibrocota ...	7

The present discussion has been almost completely confined to a consideration of rayon staple blends having a crease-resistant finish. We are applying the BFT machine to abrasion studies in other important fields, which we may instance here—a study of cotton–rayon staple blends and a study of the relative behaviour of synthetic fibres.

The BFT can be reasonably easily modified to give an additional flat abrasive motion such as is required in a universal instrument, and this BFT Mark IV exists in prototype.

* * *

The experimental work described in this paper has been considerably helped by the labours of a number of colleagues in the Courtaulds organisation: at Bocking, the members of the Textile Research Laboratory and the Bocking Mill staff have processed and produced the fabrics examined; the Use–Development Organisation under Dr. H. A. Thomas has been responsible for the design of fabrics and their observation in field-trials; and members of the staff of the Droylsden Laboratory have helped in general discussions and comparison of various machines. To all of them we offer our thanks.

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(*MS. received 14th April 1955*)

Note added in Proof

FORMAL DERIVATION OF DUTY FACTOR

Since the manuscript of this paper was prepared, Mr. F. I. Musk of the Operational Research Department, Courtaulds Ltd., Coventry, has suggested to us privately that the duty factor can be derived from the data of Table III by the methods of Discriminatory Analysis. We have treated the data along the lines indicated, and we present here a simplified abstract of the findings.

Discriminatory Analysis is a statistical technique which defines the line, quadratic or cubic, which separates two sets of points in a plane; or the plane, quadratic, or higher surface which separates two sets of points in space. The method can be applied to hyperspace of any number of dimensions.

In the present instance the problem is to define the curve which best separates the unsatisfactory from the satisfactory set of points when B is plotted against P. An inspection of the plot shows at once that no straight line will be satisfactory. The generalised quadratic is next to be tested—

$$aB^2 + bP^2 + BP + cB + dP + e = 0$$

A sketch of the curve indicates that it probably becomes asymptotic to small finite values of P and B when B and P respectively become very great; a and b may therefore be set at zero, and the equation to be examined reduces to the simple hyperbola—

$$BP + cB + dP + e = 0$$

The data of Table III are consistent with zero or small negative values for e. Since it is always desirable to avoid the use of unjustifiable constants, e will be set at zero. A series of boundary curves between the two sets of points, consistent with the data, can now be drawn between the limits defined by the equations—

$$BP - 2{\cdot}5\,B - 3{\cdot}0\,P = 0$$
$$BP - 3{\cdot}0\,B - 2{\cdot}5\,P = 0$$

The best curve for the boundary between the two sets is thus—

$$BP - 2{\cdot}75\,B - 2{\cdot}75\,P = 0$$

which may be written—

$$\frac{2BP}{B+P} = 5{\cdot}5$$

The boundary is therefore the curve of B against P for a constant duty factor of 5·5.

Discriminatory Analysis thus elegantly confirms the definition of duty factor, already arrived at empirically as—

$$D = \frac{2BP}{B+P}$$

We may note here that if differing arbitrary test conditions (chiefly of machine weighting) and normalising factors had been chosen, then a parallel analysis would, in general, have shown a weighted rather than a simple harmonic mean to be the proper expression for the duty factor.

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Discussion

Mr. R. E. HUNT: Do results obtained by Dr. Morton substantiate Schiefer's findings of apparent cross-over in abrasion results with variation in applied load in the case of untreated and resin-treated viscose rayon?

Dr. MORTON: We have not studied cross-over effects in detail, since they are, in any case, of second-order importance. They do not occur in flexing, but may, in certain series of pure-finished fabrics, be found in the ball test, conducted at varying head loads. The published results of Nuessle[9] suggest that cross-over is virtually absent at all reasonable resin contents; since the duty factor is confined to resin-finished fabrics, we anticipate that the interpretation of the data is in no way complicated by such cross-overs.

Dr. F. C. WOOD: Would not any function of P and B, say—

$$\frac{aPB}{\beta P+\gamma B}$$

where α, β, γ are arbitrarily chosen, give a more convenient scale of duty factors, rather like the nautical Beaufort wind scale?

Dr. MORTON: We are sure, by empirical testing of the data, that the harmonic mean is a better diagnostic figure than a weighted harmonic mean ($\beta \neq \gamma$ in Dr. Wood's equation). No simple function of B and P, other than a harmonic mean, appears to have any advantage in the derivation of a figure of merit, presumably since this mean is already extremely sensitive to a low value in either controlling factor.

Mr. H. A. TURNER: Although the ball and the flexing test may not show it, are not flat abrasion tests greatly influenced by the presence of resin deposits external to the fibre? As an extreme example, I remember a service fabric—a 14-oz. duck stiffened with urea–formaldehyde or similar resins—which, when tested on the Martindale machine, simply would not rub through, provided that a sharp abrasive was excluded. The resin seemed to cement the whole structure in a mass and to encourage the formation of a hard smooth surface on which a fabric used as abradant slipped with very little destructive effect.

Dr. MORTON: Our results show that many resin treatments (e.g. the methacrylate addition in Fig. 7) improve flat abrasion results. Cementing of fibres together will tend to reduce the flex-test figure catastrophically and to increase the ball penetration test value.

Mr. W. J. ROFF: What is the minimum resin content that has been observed to produce a low duty factor?

Dr. MORTON: We would not be inclined to apply the duty factor method to fabrics containing less than 3–4% of reasonably wash-fast resins. From 3% to, say, 18% of resin, careful application can yield a fabric with an unimpaired duty factor; unwise application can give low results over the same range.

Dr. H. R. RICHARDS: Is there any correlation between the duty factor and the amount of resin applied?

Dr. MORTON: The duty factor of fabrics with a very heavy addition of resin is apt to be low, but there are many other factors involved, notably embrittlement by overpolymerisation and careless use of stiffening agents.

Dr. RICHARDS: Does the use of softeners affect the duty factor?

Dr. MORTON: Addition of lubricants can improve the duty factor in certain flex-sensitive fabrics; this is correlated with an improvement in the wear properties of these fabrics.

Mr. J. BOULTON: There are many factors other than resin content which affect abrasion. A large proportion of unpolymerised resin to give a weighted but not wash-fast fabric may produce one result; high acidity of catalysis to give a resin which hardens with minimum baking may yield a quite different abrasion result. Neither is resistant to creasing.

Mr. H. E. BLAKELEY: Will the new abrasion machine be available to the trade?

Dr. MORTON: We have already made arrangements with R. A. Jones & Sons Ltd., 76 and 78 High Street, Southend-on-Sea, for the production and sale of exact replicas of the BFT Mark III machine. Calibration of the machines will be certified by the Bocking Laboratory.

COMMUNICATIONS

Bleaching Jute with Hydrogen Peroxide

H. CHATTERJEE and K. B. PAL

A single-bath process of bleaching jute with hydrogen peroxide is described with particular reference to loss of weight, lignin, and breaking strength as well as the colour of the bleached material and the composition of the residual lignin. The influence of variables— e.g. reaction temperature, time, bleach concentration, liquor ratio, pH, bleachability with peroxide compared with chlorite— has been studied.

Jute may be bleached white with peroxide with only a small loss of lignin, and the composition of its residual lignin remains more or less unchanged. It is believed that the point of attack by peroxide is a phenolic hydroxyl group and that the colour change is mainly due to an unknown structural modification of native lignin. A one-volume bath of peroxide containing a stabiliser under alkaline conditions, and a 1% solution of acidified chlorite at a liquor ratio of 10 : 1 and a temperature of 60–70°C., are suitable for bleaching jute. In both cases over 70% of the yarn strength can be maintained in the fully wet condition. The tendency of the bleached material to become yellow on storage is much less pronounced with peroxide than with hypochlorite or chlorite. The quality of such a yarn is unimpaired by long storage.

INTRODUCTION

Surveys of the literature on textile bleaching with hydrogen peroxide as well as its relation to other bleaching agents have been published in recent years by the AATCC [1] and by Laporte Chemicals Ltd.[2]. It is generally known that for cotton goods the peroxide liquor should have a strength of 0·5–1·0 volume, should be used under alkaline conditions at a temperature of 80–85°C., and should contain a stabiliser, such as sodium silicate, magnesium sulphate, a phosphate, etc. As a preliminary step, the cotton goods are well "bottomed", i.e. scoured with alkaline liquor alone or with a small concentration of peroxide. But this process cannot be applied to jute and allied long fibres for the following reasons. The jute filament is an aggregate of multiple fibre elements about $\frac{1}{8}$ in. long, embedded in and cemented together by a matrix of hemicellulose and lignin. The strength of a fibre depends primarily on the bundle of the molecular chains of cellulose. In jute, the fibre elements are short, and the cementing material contributes greatly to the strength. In any wet finishing process this material is readily attacked, the fibre elements become swollen and are loosened, and the fibre thus becomes weaker. Besides, jute goods, unlike cotton goods, contain about 5% of mineral oil, added for the spinning operation. For desirable results to be obtained on bleaching, this oil as well as the natural fat and wax should be removed, not by conventional scouring, which is detrimental to jute, but by extraction of the material with an organic solvent, e.g. carbon tetrachloride, trichloroethylene, benzine, etc., as described by Fort [3] and Garrett [4]. Otherwise it is desirable to use a wetting agent, e.g. Lubrol (ICI), in the bleaching bath. The conditions of bleaching should be adjusted to aim at a reasonable compromise between the degree of brightening and the residual strength.

Bleaching processes for flax and jute using hot alkaline peroxide following hypochlorite have already been reported by several investigators [5–11]. Parsons [12] suggested the possibility of bleaching jute with peroxide alone to get the best results. Preliminary observations on bleaching jute throughout with hydrogen peroxide have also been reported from these laboratories. It was claimed that peroxide bleaching was the least degrading, that very little lignin was lost, and that the colour of the bleached material was good [13]. Subsequently several investigators [14, 15] published data which supported these findings. The object of the present paper is to place on record the detailed results obtained here.

EXPERIMENTAL

Clean jute fibre was cut into short lengths, well mixed, and extracted in a Soxhlet for 6 hr. with alcohol–benzene (1 : 1 by volume). It was then dried at room temperature, and 2-g. samples were weighed out and dried to constant weight at 105°C. to determine their moisture content. Eleven-inch lengths were cut from the middle portion of each reed of jute, and each was divided into two halves, two lots being thus obtained. These were combed in the same manner and collected into two bundles, one of which was intended for treatment. This was conditioned at a standard temperature and humidity, and its weight recorded before and after treatment to obtain the loss of weight. Both lots were tested for the ballistic work of rupture by the twisted-bundle method [16].

The jute yarns used were of average quality with 9–10 lb. grist (i.e. weight of 14,400 yd.). Generally, each skein, 90 in. long, with 240 threads, was marked into three equal parts by being tied twice at each mark to maintain the twist, and was cut between adjacent knots into triplicate bundles. These were also solvent-extracted, conditioned, and weighed before and after treatment, as were the fibre bundles. Single-thread strength was determined on the conditioned material, both treated and untreated. The same test length (18 in.) of about 240 threads constituting a bundle was broken. Replicate tests were made to check the results.

The materials were treated in beakers with the requisite amount of solution and stirred carefully from time to time during the treatment. For higher reaction temperatures the beakers were covered and placed in a hot water-bath. After treatment the materials were washed, antichlored when required, and washed thoroughly, the

TABLE I

Effect of Time of Bleaching on Loss of Weight, Loss of Lignin, Lignin Methoxyl Content, and Colour of Fibre

(1-vol. H_2O_2 soln.)

Time of Treatment (hr.)	Loss of Weight*	Lignin Residual*	Lignin O·CH_3†	Loss of Lignin*	Colour
0	0	11·6	17·6	0	Grey
½	5·8	10·4	17·0	1·2	Yellowish
1	6·9	10·3	17·1	1·3	Almost white
1½	6·4	10·0	—	1·6	White
2	7·5	9·7	17·4	1·9	White
2½	7·6	9·2	—	2·4	White
3	7·6	9·6	17·1	2·0	White
4	7·5	9·3	17·0	2·3	White
5	7·6	9·3	17·8	2·3	White
6	7·7	9·0	—	2·6	White

* Expressed on 100 g. of oven-dry jute
† % on weight of lignin

liquid being pressed out of the materials, which were then dried at room temperature. Great care had to be taken throughout the process not to damage the samples for strength tests. The small fibre samples after treatment were filtered through tared glass crucibles for chemical analysis.

The concentration of bleach liquor was determined iodimetrically, and the pH by means of the B.D.H. universal indicator. Residual lignin was estimated by the 72% sulphuric acid method on the test fragments after strength tests had been made, and the methoxyl value of the residual lignin was determined by the modified Vieböck and Schwappach method [17].

TABLE II

Effect of Concentration of Hydrogen Peroxide on Loss of Weight and Loss of Lignin

H_2O_2 Concn. (vol.)	Time of Treatment (hr.)	Loss of Weight*	Residual Lignin*	Loss of Lignin*
0	0	0	11·6	0
1	2	7·5	9·7	1·9
	6	7·7	9·0	2·6
4	2	8·2	9·1	2·5
	6	8·4	9·0	2·6
8	2	8·1	9·1	2·5
	6	7·6	9·0	2·6
105	2	8·2	8·2	3·4

* Expressed on 100 g. of oven-dry jute

RESULTS

A series of exploratory experiments showed that jute could not be bleached by a single steep in hydrogen peroxide if the strength of the latter was less than 0·5 volume and its pH was below 8.

Effect of Peroxide Bleaching on Fibre

Two-gram samples of cut fibre were treated at 65–70°c. with 100 c.c. of peroxide varying in strength from 1 to 105 volumes and containing 1 g. sodium silicate (140°Tw.) and 0·5 g. sodium orthophosphate, the pH being 10–11. For thorough wetting and treatment in loose form, a long liquor ratio (50 : 1) was chosen. Standard bundles of filaments and yarns were, however, treated in much shorter liquor ratios with low bleach concentration to maintain the residual strength at a fairly high level. The results are given in Tables I–IV.

Effect of Peroxide Bleaching on Yarn

Single-steep peroxide bleaching of yarn was examined with regard to various effects of reaction temperature, time, pH, liquor ratio, etc., the concentration of peroxide bleach being kept constant at 1 volume strength with 10 g. silicate plus 5 g. phosphate per litre or with some other buffer system. The results are given in Tables V–VIII.

TABLE III

Effect of Hydrogen Peroxide Bleaching on Loss of Weight and Strength of Jute Filament

(One lot of combed fibre 11 in. long was bleached, and the duplicate served as the control)

Ref. No.	Treatment	Loss of Wt. on Conditioned Jute (%)	Ballistic Work of Rupture (in.–lb.)* Wet	Residual Strength (%) Wet	Colour
690	Control, extracted with alcoholic benzene	0	29·5	100	—
691	1-vol. H_2O_2 containing 10 g. silicate + 5 g. phosphate per litre, liquor ratio 10 : 1, 60–65°c., 4 hr.	2·9	25·9	88	White
692	Control, extracted with alcoholic benzene	0	36·1	100	—
693	Bleached as 691 but at 20 : 1, 25°c., 24 hr.	3·5	29·9	83	Slightly inferior to 691

* Mean of 28–30 observations— standard error within ±3%. Corrected for loss of weight

TABLE IV

Effect of Alkali Boiling and Hydrogen Peroxide Bleaching on Lignin Content of Jute Fibre

	Treatment	Lignin Residual*	Lignin Loss*
(*a*)	Untreated	15·0	0
(*b*)	Boiled with 0·5% NaOH for 4 hr.	12·3	2·7
(*c*)	Bleached with 1-vol. H_2O_2 for 2 hr.	12·5	2·5
(*d*)	(*b*) bleached as (*c*), colour yellow	11·9	3·1
(*a*)	Untreated	14·3	0
(*b*)	Boiled with 1% NaOH for 4 hr.	12·3	2·0
(*c*)	Bleached with H_2O_2 as (*c*)	12·3	2·0
(*d*)	(*b*) bleached as (*c*), colour yellow	11·5	2·8
(*a*)	Untreated	13·2	0
(*b*)	Boiled with 0·5% NaOH for 4 hr.	10·0	3·2
(*c*)	Bleached with H_2O_2 as (*c*)	10·4	2·8
(*d*)	(*b*) bleached as (*c*), colour yellow	9·2	4·0

* Expressed on 100 g. of oven-dry jute

TABLE V

Effect of Temperature on the Peroxide Bleaching of Jute Yarn

(Liquor ratio 10 : 1. Time 2 hr.)

Reaction Temp. (°C.)	Breaking Load* (lb.) (Single thread) Wet	Residual Strength (%)	Colour
Control	7·8	100	—
40–50	5·9	76	Yellowish white
55–60	5·7	73	Yellowish white
Control	7·5	100	—
65–70	5·5	73	Almost white
75–80	5·4	72	Almost white
Control	7·3	100	—
85–90	4·9	67	Almost white
95–100	4·7	64	White
Control	9·6	100	—
32–34 (18 hr.)	7·4	77	Almost white

* Mean of about 240 breaks. Standard error within 2%

TABLE VI

Effect of Reaction Time on Hydrogen Peroxide Bleaching of Jute Yarn

(Liquor ratio 10 : 1. Temp. 65–70°C.)

Reaction Time (hr.)	Breaking Load* (lb.) (Single thread) Wet	Residual Strength (%)	Colour	Final Strength of H_2O_2 (vol.)
Control	8·8	100	—	1
1	6·8	77	Almost white	0·52
2	6·6	75	Almost white	0·46
Control	9·0	100	—	1
3	6·7	74	Almost white	0·47
4	6·8	76	Almost white	0·37
Control	9·1	100	—	1
5	6·5	71	Almost white	0·40
6	6·4	70	Almost white	0·34

* Standard error within 1·2%

TABLE VII

Effect of pH and Buffer System on Hydrogen Peroxide Bleaching of Jute Yarn

(Liquor ratio 10 : 1. 2 hr. at 65–70°C.)

Ref. No.	pH	Buffer System	Residual Strength (%)* Wet	Colour	Final Strength of H_2O_2 (vol.)
932	7	Unbuffered	92	Slight brightening	0·85
993	7	Citric acid + Na_2HPO_4	83	Better than 932	0·70
994	8	Citric acid + Na_2HPO_4	80	Yellowish white	0·60
925	8	H_3BO_3 + KCl + NaOH	77	As 993	0·80
926	9	H_3BO_3 + KCl + NaOH	82	As 994	0·60
929	10	H_3BO_3 + KCl + NaOH	81	As 932	0·10
931	10	$NaHCO_3$ + Na_2CO_3	83	As 932	0
911	10	Silicate + phosphate	75	Almost white	0·46

* Standard error within 1·2%

TABLE VIII

Effect of Liquor Ratio on Hydrogen Peroxide Bleaching of Jute Yarn

Liquor Ratio	Breaking Load* (lb.) (Single thread) Wet	Residual Strength (%)	Colour
	18–24 hr. at 32–34°C.		
Control	9·6	100	—
10 : 1	7·4	77	Almost white
Control	8·8	100	—
20 : 1	5·9	67	White
	2 hr. at 80–85°C.		
Control	7·3	100	—
10 : 1	4·9	68	Almost white
Control	6·1	100	—
20 : 1	3·3	54	White

* Standard error within 2%

Effect of Storage on Colour and Strength of Bleached Jute Yarn

Examination of test samples bleached with chlorite, hypochlorite, and hydrogen peroxide, and stored under similar conditions for about a year, revealed that the tendency to yellowing was very much less pronounced with the peroxide-treated samples, as expected. A considerable amount of the low-molecular hemicelluloses which may contribute to yellowing[18] had already been removed by the alkaline peroxide treatment. A jute sample bleached to full white with chlorite and kept under absolute alcohol in a sealed test tube, which has been exposed to daylight at a window for several years now, is still unchanged in colour.

To ascertain the effect on strength, replicate cut-skein samples of a yarn were bleached to a pale yellow with sodium chlorite (1·1%, acidified with acetic acid), at a liquor ratio of 20 : 1 and pH 5, on a boiling water bath for 1 hr., antichlored with sulphurous acid, and washed. They were dried at room temperature. One was conditioned and tested for strength. The others were kept hanging on a stretched string in a store room with a door open during the day and a fanlight open day and night. The samples were withdrawn from storage at intervals and tested for strength (Table X).

DISCUSSION OF RESULTS

From the results given in Tables I, II, and VI, it is evident that bleaching with peroxide is practically complete in two hours with 1-volume strength at 65–70°C. The material loses only about 2% of its weight of lignin. The bleach liquor suffers about 50% exhaustion (Table VI). The difference between the loss of weight and the loss of lignin is accounted for by hemicelluloses, 5–6%, which are lost in the hot alkaline bleach. A parallel set of experiments without peroxide in the treating liquor showed that there was no loss of lignin due to the alkaline medium. By over-bleaching, a little more lignin can be removed, but at the cost of the strength of the material and without appreciable improvement in colour.

The peroxide-susceptible fraction of lignin, 2–3% (Tables I and II), is probably differently constituted from the rest. Presumably it was the same fraction which Sarkar and co-workers[19] found to be dissolved away from jute fibre by boiling 0·25–2·0% caustic soda solution. The results of Table IV support this.

Table V demonstrates that peroxide bleaching of jute yarn at 60–80°C. for 2 hr. or at 32–34°C. overnight is satisfactory with respect to both colour and maintenance of strength (70%) in the fully wet condition. A hotter bleach seems to cause greater weakening.

It will be seen from the results of Table VII that jute cannot be bleached with peroxide if the pH of the peroxide bath is below the alkaline range. Even at pH 9–10 maintained with common buffer salts, the bleaching action of hydrogen peroxide is unsatisfactory without the addition of a suitable stabiliser and much peroxide is lost. But the bath containing the silicate–phosphate mixture, which not only acts as a stabiliser for peroxide but also maintains the proper pH, is quite efficient.

Table VIII shows that no advantage is gained by using a peroxide bath of 1 volume strength at a higher liquor ratio than 10 : 1.

TABLE IX

Bleachability of Jute— Hydrogen Peroxide *versus* Sodium Chlorite

Treatment	Approx. Loss of Wt. (%)	Breaking Load* (lb.) (Single thread) Wet	Residual Strength (%)	Residual Lignin (%)	Colour
Control	0	9·9	100	11·1	Grey
0·7% Chlorite†	5·2	7·2	73	8·0	Yellowish white
1-vol. H_2O_2‡	7·8	7·3	74	10·3	Almost white
Control	0	9·0	100	13·2	Light brown
0·7% Chlorite†	6·3	7·2	80	9·6	Yellowish white
1-vol. H_2O_2‡	7·8	6·8	76	12·4	Almost white
Control	0	6·9	100	13·6	Light grey
0·7% Chlorite†	4·3	5·3	77	9·7	Cream
1-vol. H_2O_2‡	7·8	5·3	77	12·3	Almost white

* Standard error within 1·3%

† 0·7% Chlorite + acetic acid, pH 3–4, liquor ratio 10 : 1, 2 hr. at 60–70°C.

‡ 1-vol. H_2O_2, pH 10–11, liquor ratio 10 : 1, 2 hr. at 60–70°C.

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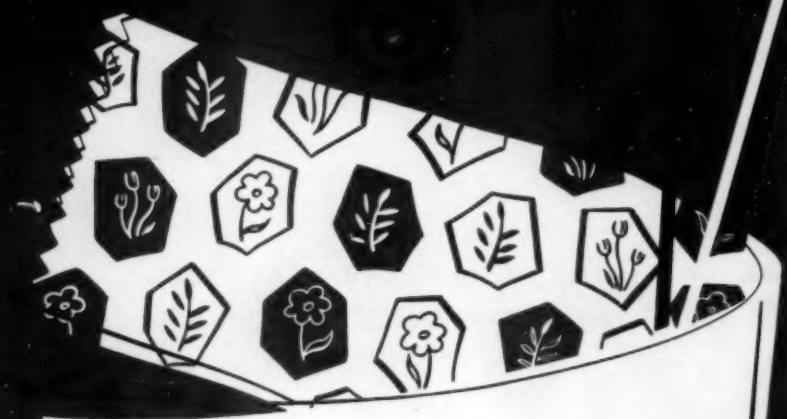

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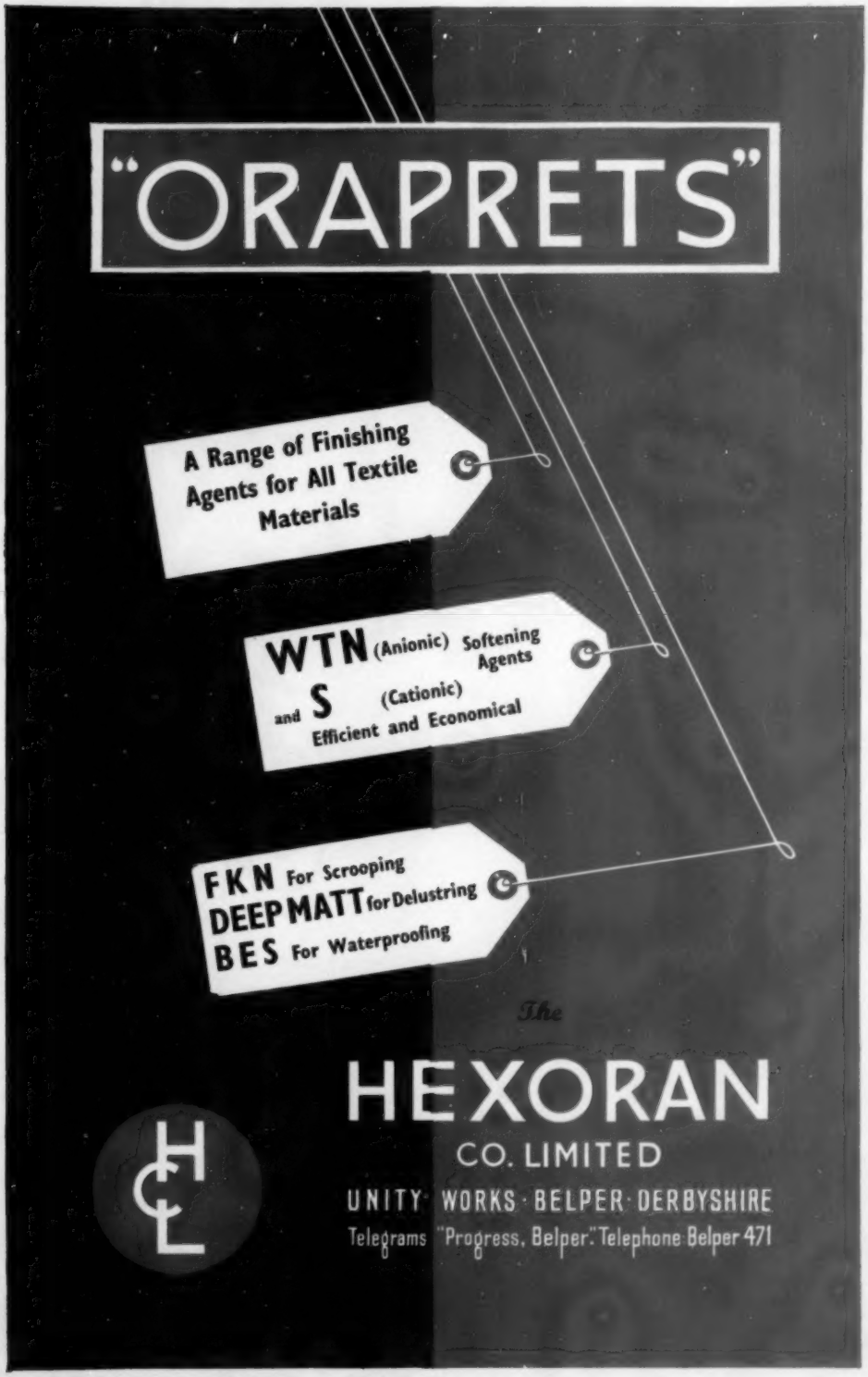
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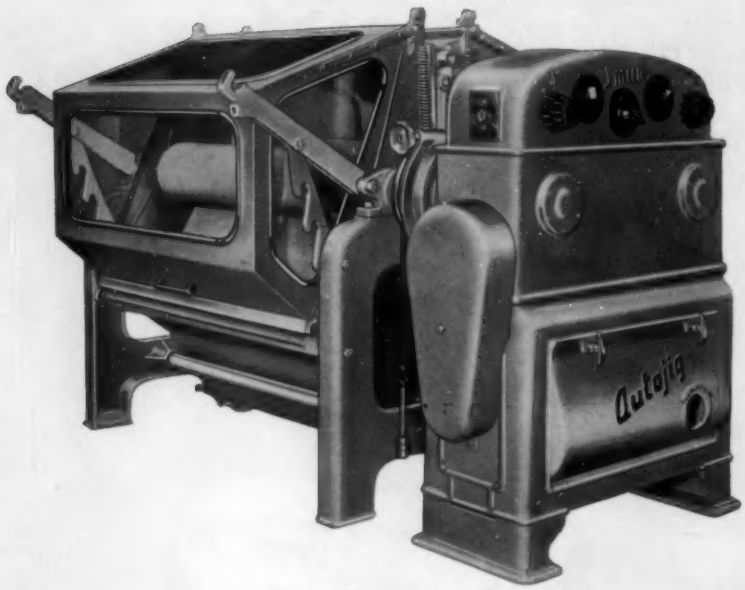

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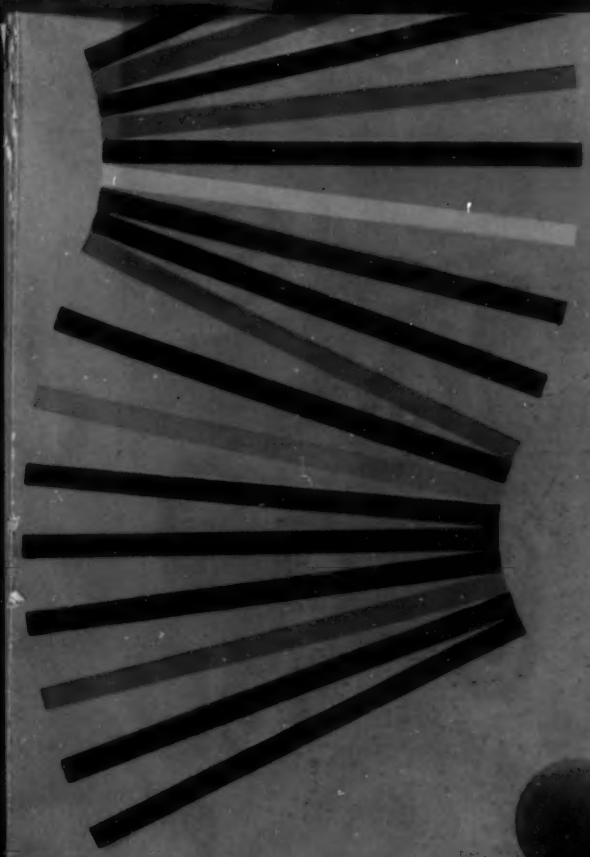

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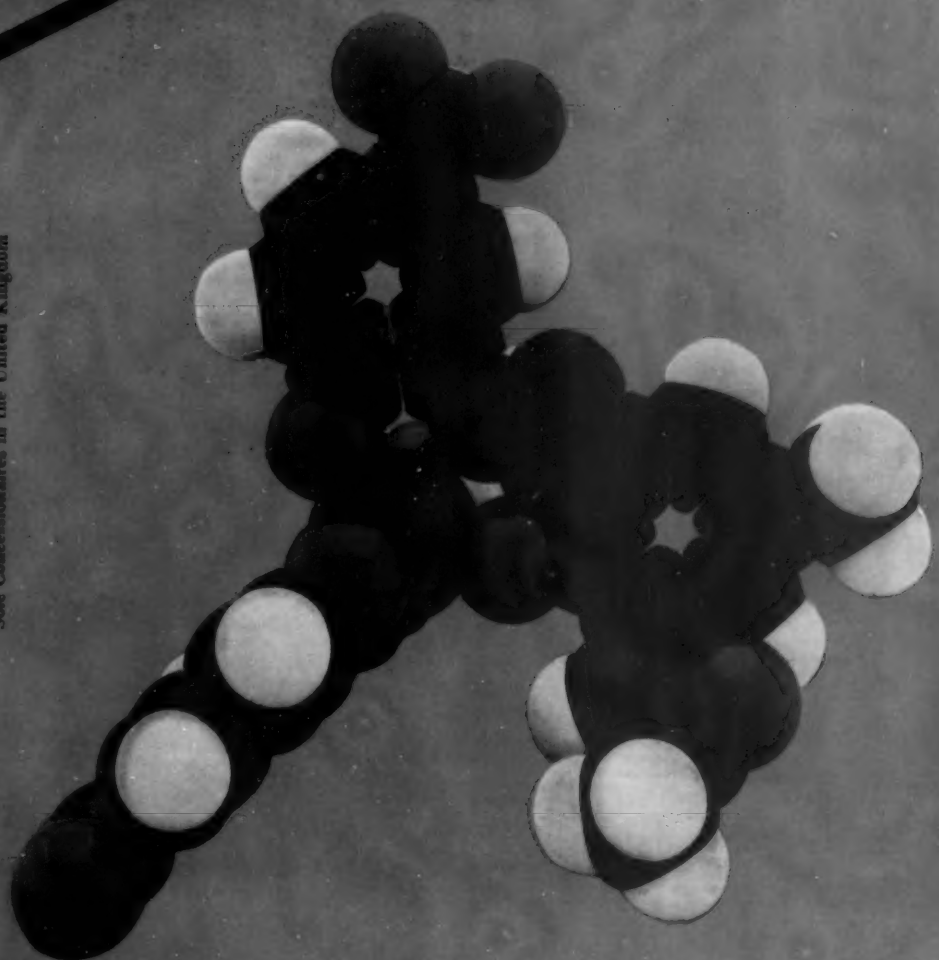

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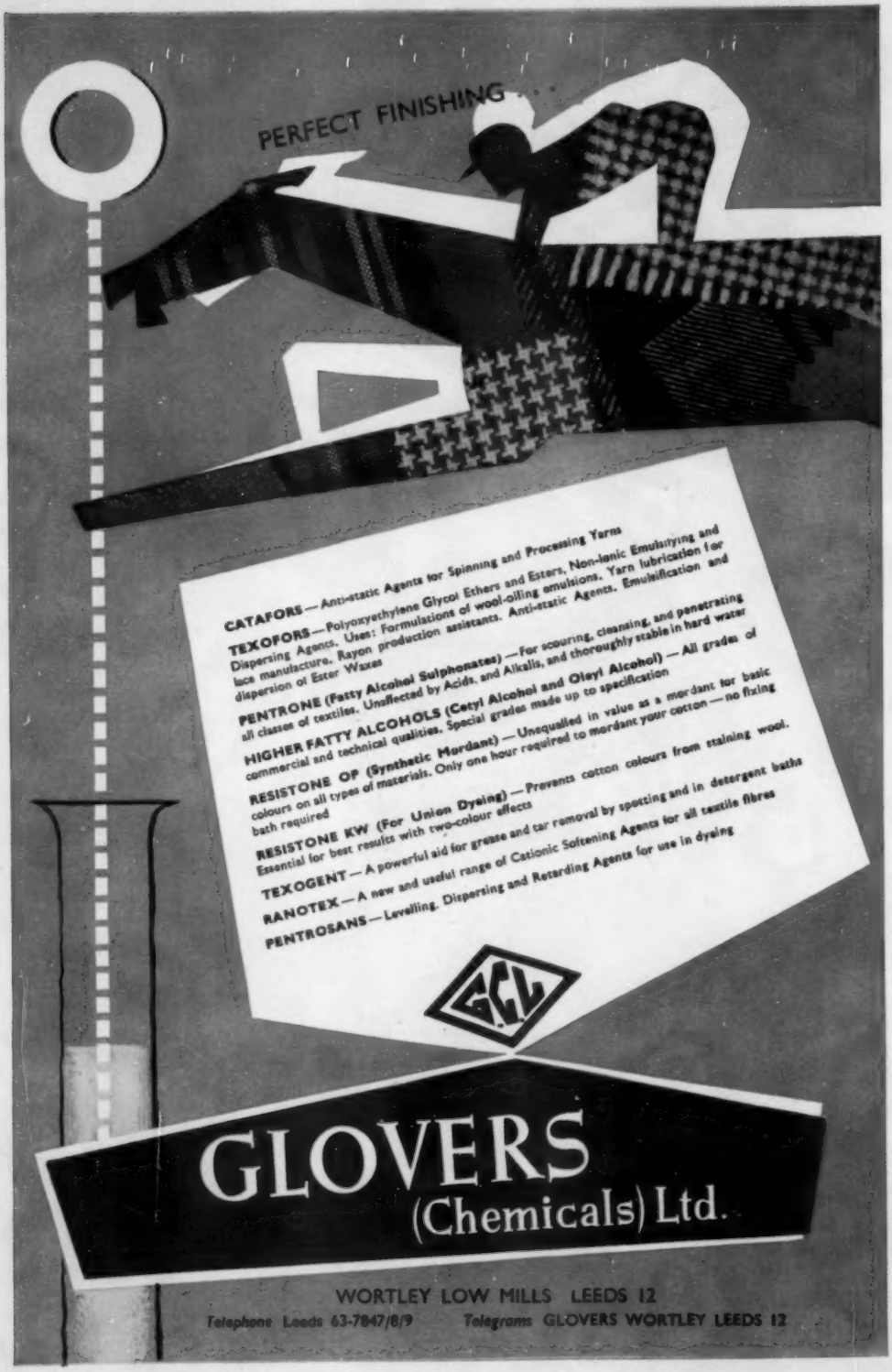
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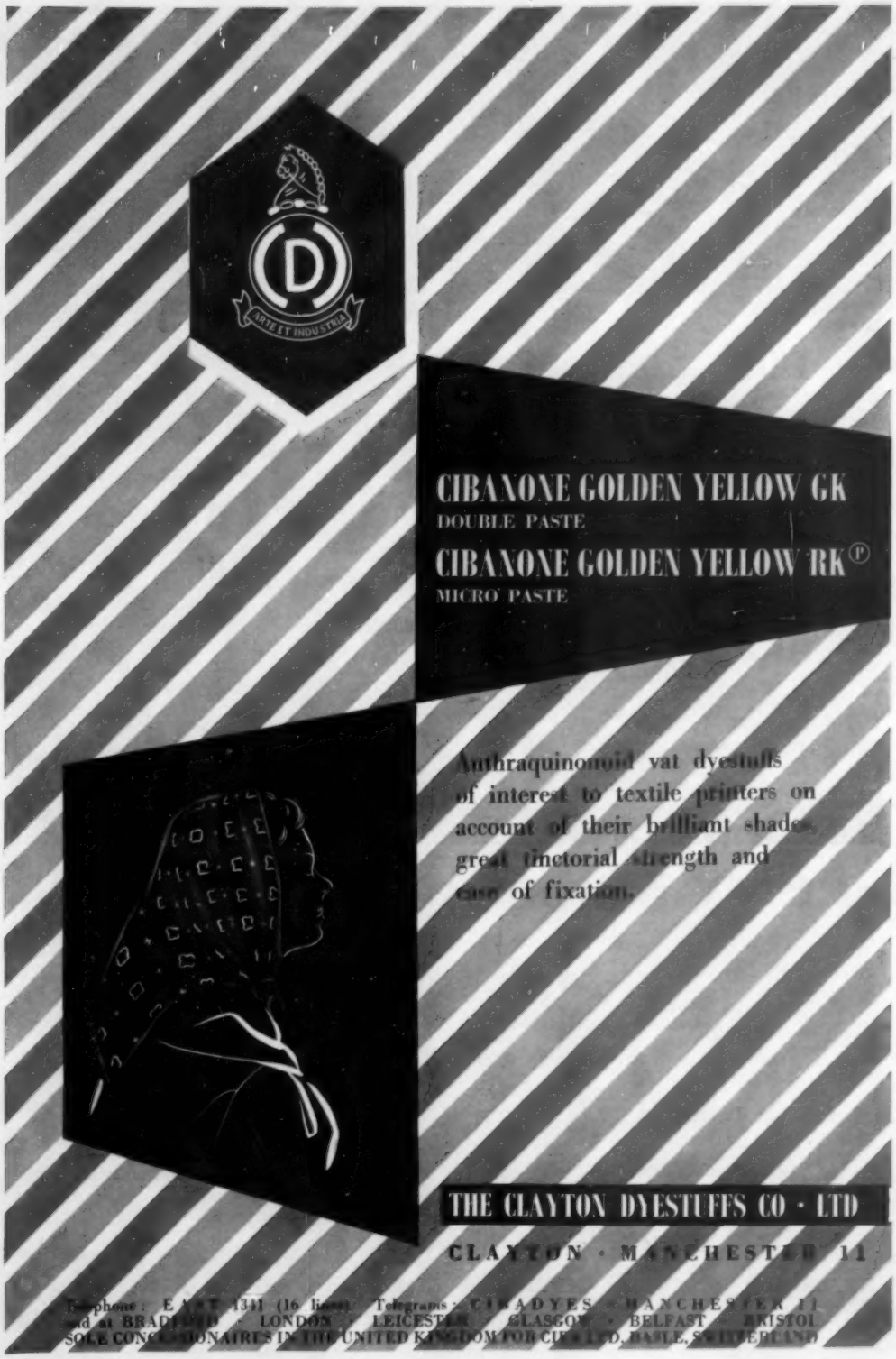
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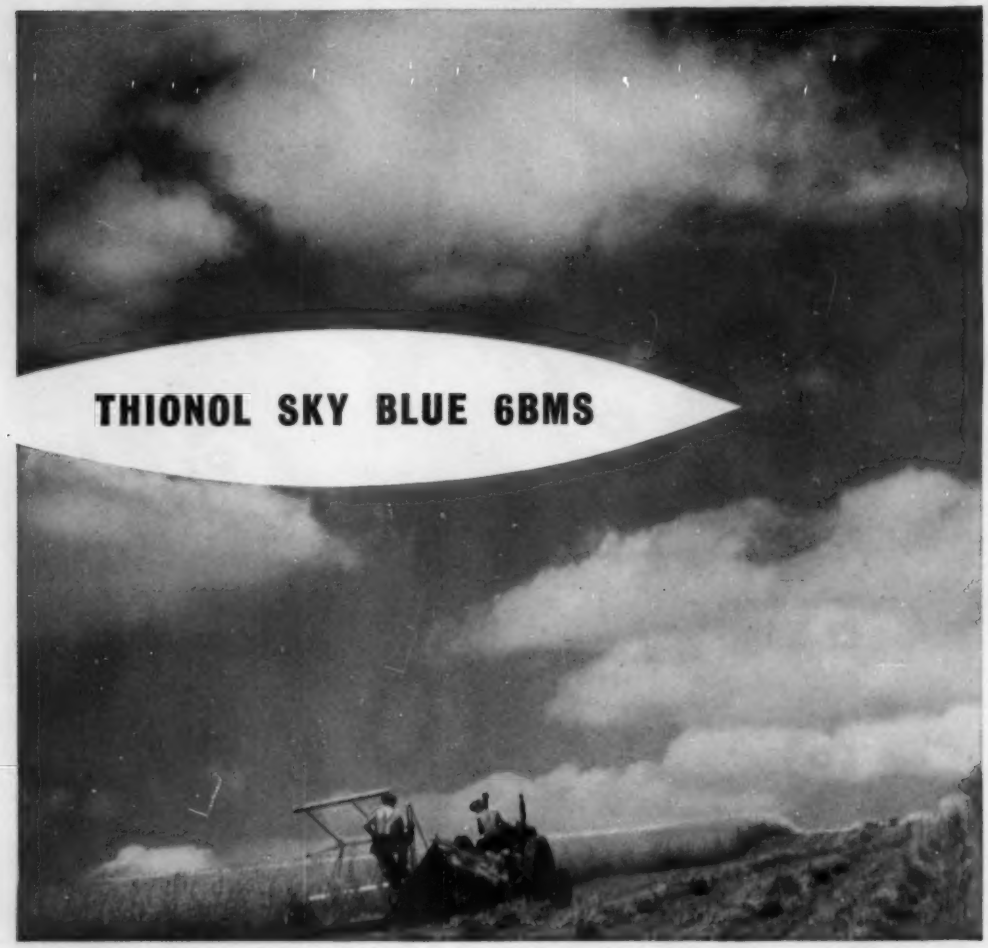

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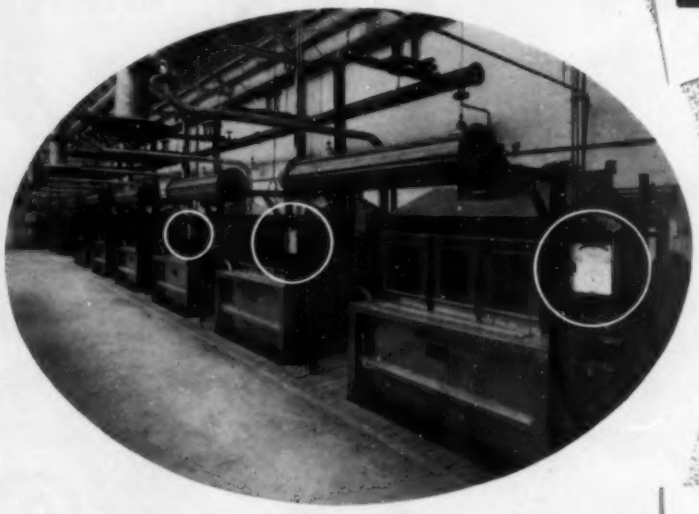

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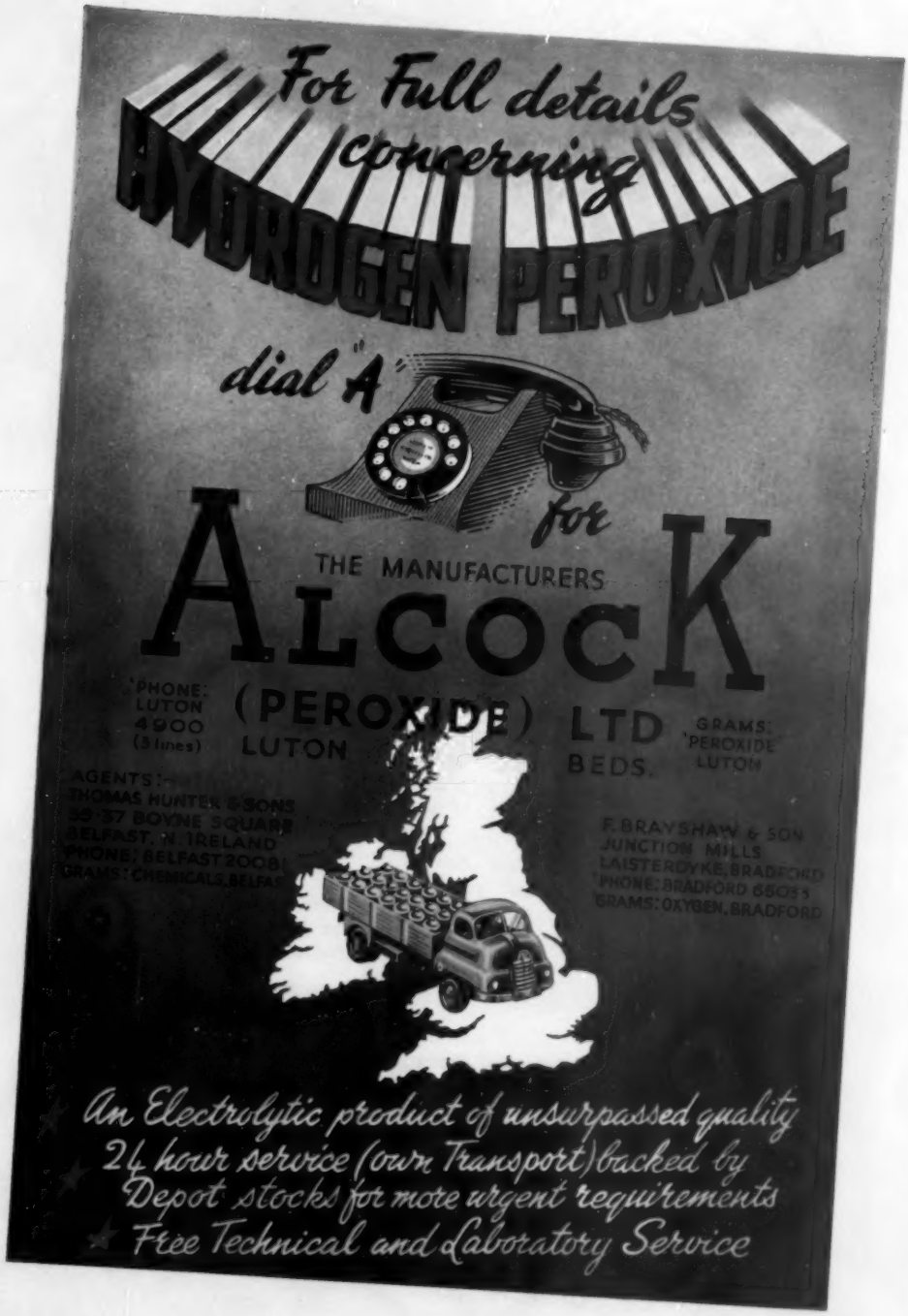
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CONTENTS

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SULPHUR DYES
ANTHRAQUINONE DYES AND INTERMEDIATES
INDIGOID DYES
PHTHALOCYANINE PIGMENTS
PHTHALOCYANINE DYES
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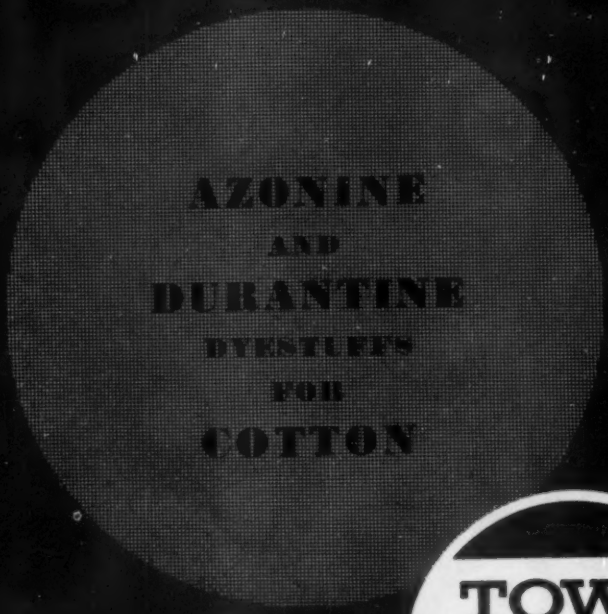

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It is noteworthy (Table IX) that over 70% of the wet strength of three yarns of different quality can be maintained by bleaching with acidified sodium chlorite or with peroxide. The peroxide treatment, being alkaline, gives a better and more lasting colour as found by storage experiments, but all specks are not killed.

The loss of strength and weight with peroxide treatment is mainly due to partial removal of hemicellulose, whereas with chlorite it is mainly due to removal of lignin. This was first observed by Ridge, Little, and Wharton [6]. The lignin content of the peroxide-bleached yarn is less than that of the original by only about 1%.

It was reported previously by Sarkar and Chatterjee [20] that even a mild alkaline treatment before bleaching jute with chlorite promotes loss of strength and of weight and should be avoided. The same is true for peroxide bleaching also, because of greater loss of hemicellulose.

TABLE X

Effect of Storage on Strength and Quality of Bleached Jute Yarn

Time of Storage (days)	Breaking Load* (lb.) (Single thread) Air-dry	Quality Ratio†
0	8·9	94
31	9·0	92
111	8·8	95
157	8·7	92
406	8·5	90
458	9·1	98
550	8·0	84
613	6·9	74
682	7·6	81
715	8·1	87
788	7·3	72

* Standard error within 1·5%

† The term *quality ratio* is defined as the breaking load divided by the grist (i.e. weight of 14,400 yd. of yarn), both in lb. × 100

From the results presented in Table X, it is important to note that the strength of the bleached yarn remains practically unchanged for 15 months of storage, after which, however, loss of strength by 10–15% takes place in a further 8 months' time.

ORIGIN OF THE COLOUR OF RAW JUTE

Although jute is bleached white by hydrogen peroxide and most of the lignin remains in the bleached fibre (the lignin content is about 1% less than that of raw jute, Table IX), the residual lignin appears to be coloured like the original when isolated by the 72% sulphuric acid method. Table I shows that its composition with respect to the most important functional group, viz. methoxyl, is not changed to any appreciable extent by peroxide treatment. The isolated lignin is not bleached, but a major portion is dissolved. On the other hand, alkali lignin separated from a caustic soda extract of jute, which also is coloured, is bleached to a colourless product with a small white residue.

On the assumption that lignin *in situ* is the cause of the colour of jute fibre and that it is a flavone type of colouring matter, we can visualise the bleaching reaction between peroxide and the lignin–hemicellulose complex at a phenolic hydroxyl group. This reaction probably brings about an orientation in the structure of the chromophoric groups [21] to give a colourless product, but does not appreciably change the chemical composition of the residual lignin.

Das and co-workers [22] cited evidence from other observations [14, 23] in support of the view that the colour of jute depends on the amount of uncombined or labile lignin, which is easily attacked by hydrogen peroxide. Native lignin, however, may be coloured or colourless in its various phases of development from the primary to the secondary stage.

Protolignin and isolated native spruce lignin become almost white on methylation with diazomethane [24]. A free phenolic hydroxyl group is first methylated. The fact that this product observed by Brauns [25], as well as the peroxide-bleached jute observed by the authors, still gives the phloroglucinol–hydrochloric acid colour reaction indicates that the enolisable carbonyl group of the unsaturated carbon side-chain of the lignin building unit is intact. Alkali-boiled jute, like lignin fully methylated with diazomethane, does not give this colour reaction, showing that the carbonyl group is fixed by these treatments. The results given in Table IV show that the alkali-treated jute, which has already lost the peroxide-susceptible fraction of lignin, is not bleached white but merely becomes yellow when treated with peroxide [14], with a little further elimination of lignin.

The methoxyl content of lignin remains more or less unchanged during the peroxide bleaching of jute (Table I). Moreover, methylation of native lignin is marked by reduced consumption of sodium peroxide [26]. It is therefore suggested that a phenolic hydroxyl group is the threshold of attack by peroxide on native jute lignin, as in the case of diazomethane on spruce lignin.

* * *

The authors' best thanks are due to Dr. P. B. Sarkar, Director of Research, for his keen interest in the work and to Shri S. B. Bandyopadhyay, Testing Superintendent, who was responsible for the physical tests.

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An Explanation of Dyeing Mechanisms in Terms of Non-polar Bonding

A. N. DERBYSHIRE and R. H. PETERS

Inadequacies are demonstrated in previous attempts to explain dyeing mechanisms on the assumption of polar forces between dye and fibre. The important part played by non-polar forces in adsorption phenomena generally is illustrated by examples. It is postulated that such forces are mainly responsible for dye–fibre attachment and that in aqueous solution they occur between hydrophobic surfaces.

The relative importance of hydrophobic and hydrophilic properties in fibres is deduced from their moisture regain figures and is discussed in relation to fibre structure. The properties of dyes are then similarly considered. Thence the dyeing behaviour of each type of fibre is predicted from the postulate that dye substantivity is due to non-polar forces. It is concluded that this assumption is sufficient to explain the methods adopted for dyeing different fibres and that it eliminates anomalies incapable of explanation on the assumption that dye–fibre attachment is due to polar forces.

Introduction

A knowledge of the forces which bind dyes to fibres is of obvious importance in the development of a theory of dyeing. In this paper the attractive forces which operate in adsorption processes will be considered in their relation to the structure and properties of different fibres. Thus it may be possible to present a unified picture of dyeing mechanisms from existing knowledge on dye substantivity.

Of all dyeing procedures, the dyeing of cellulosic fibres with direct cotton dyes has in the past been the most extensively studied [1]. This work has shown that substantive dyes of this class have linear and planar molecules containing hydrogen-bond-forming groups. It was believed that substantivity could be improved if these groups were separated by a distance which approximated to the repeat spacing of the cellobiose unit in the cellulose chain. The use of benzidine, which gives rise to this spacing, as an intermediate in the manufacture of many direct dyes was quoted as evidence in support of this assumption. However, it has recently been demonstrated [2] by construction of accurate molecular models of cellulose and of direct dyes that polar groups are so distributed along the cellulose chain that this distance has very little significance.

The dyeing of wool with acid dyes has also been investigated thoroughly [3]. Here the maximum uptake of inorganic acids such as hydrochloric acid corresponds closely with that of level-dyeing acid dyes, which shows that combination with hydrogen ions is important. It has been suggested that electrostatic forces between the dye anions and NH_3^+ groups, liberated from salt-links in the fibre by combination with hydrogen ions, make a large contribution to the binding energy. Hydrogen bonding between polar groups in the dye anion and amide groups in the wool has been postulated to account for differences in dye affinity. It has also been suggested [4] that formation of hydrogen bonds is responsible for the uptake of phenol by cellulose acetate.

Thus polar forces have been assumed almost exclusively in order to explain dyeing mechanisms. Some of the difficulties to which this assumption leads may now be considered. It must first be remembered that dyeing normally takes place from water, that is from a highly polar medium which contains both electron-donating and electron-accepting groups. In this environment polar groups in both dye and fibre will initially be strongly attached to water molecules. The formation of a dye–fibre linkage by hydrogen bonding will require a dye–water bond and a fibre–water bond both to be broken, and the freed water

molecules to be taken back into the water structure. The heat of dyeing will then be the net heat change due to all these processes, and attempts to assign it to the heat of formation of dye–fibre bonds alone can have little meaning. Recent work[5] with a number of disperse dyes has shown that their heats of dyeing on to cellulose di- and tri-acetate are similar in magnitude and opposite in sign to their heats of solution. It has also been shown[6] by direct calorimetry that the heat content of solid Naphthalene Orange G is equal to that of the same dye when adsorbed by wool. These results suggest that the forces between dye molecule and fibre in a dyeing are the same as those between dye molecules in the solid form of the dye. They are not easy to reconcile with the suggestion that hydrogen bonding takes place at specific groups in the fibre, since this cannot occur in the solid dye.

Carbolan dyes, which have a much higher affinity for wool than levelling dyes, differ from them in possessing a long aliphatic chain, to which the increase in affinity must be attributed. The formation of polar bonds cannot explain this increased substantivity conferred by a non-polar grouping. Certain vat dyes have high affinity for cellulose in the reduced form and yet contain only two O^- groups which are capable of forming polar bonds. One would expect that these solubilising groups would be predominantly attracted to the aqueous phase. Here, then, is an example of dyes which are substantive and yet contain no groups likely to form polar bonds with the fibre. The view that the O^- groups are inadequate is justified by the negligible affinity for cellulose of smaller molecules of the same type, such as anthraquinone[7]. Finally, Terylene polyester fibre will adsorb a number of organic substances such as diphenyl, which are used as carriers in the dyeing of this fibre and which contain no groups capable of hydrogen bond formation.

These deficiencies in the hydrogen-bonding theory suggest that a consideration of other bonding forces is essential and that non-polar rather than polar forces may predominate in dye–fibre attachment. Recently this point of view has received increasing support. Meggy[8], when considering the affinities of acid dyes for wool, made the following interesting suggestion as to the source of the free energy change. He postulated that, in forming an aqueous solution, the large hydrophobic surface of the dye molecule caused a separation of water molecules against their mutual attraction and so required an uptake of energy. The removal of dye through adsorption by wool will lead to the recovery of this energy by recombination of water molecules, and it will then appear as the free energy of dyeing. Calculation showed the energy available to be of the right order of magnitude to account for the measured affinities. Work has recently been done[9] on the change of molecular area of monolayers of surface-active compounds containing hydrogen-bonding groups on solutions of sucrose, glucose, and cellobiose. From the results it was concluded that the hydroxyl groups of these latter compounds, when in an aqueous environment, are too strongly attached to water molecules to be available for forming hydrogen bonds with other compounds. For this reason it was suggested that bonding of direct dyes to cellulose was due largely to non-polar van der Waals forces.

The importance of non-polar forces in the field of adsorption phenomena generally is fully recognised, and calculation shows that they are responsible for a large proportion of the heat of adsorption. Therefore, it is worth considering at greater length the rôles of all types of bonding forces which are known to take part in adsorption processes.

Possible Adsorption Forces

Consideration of various adsorption phenomena has led to the following classification of attractive forces[10]—

(1) Electrostatic attraction between charged sites in the substrate and ionic substances
(2) Attraction by induction between ionic substances and a non-conducting substrate
(3) Polar van der Waals forces (e.g. hydrogen bonds)
(4) Non-polar van der Waals forces
(5) Chemical forces.

In most adsorption phenomena some or all of these forces will contribute to the total attraction, but the classification is of value since in simple cases it is possible to calculate theoretically the contribution from each type to the total heat of adsorption. Reasonable agreement with the measured value is then found. The results[10] of two such calculations may be quoted—

(1) Adsorption Energy of Carbon Dioxide on Potassium Iodide

	kcal./mole
Non-polar van der Waals forces	3·30
Electrostatic polarisation	1·14
Dipole forces	3·25
Total	7·69
The measured adsorption energy is	7·45

(2) Adsorption of Phenol on Sodium Chloride

Non-polar van der Waals forces of C_6H_5 group	3·4
Dipole forces of OH group	5·3
Non-polar van der Waals forces of OH group	0·60
Electrostatic polarisation of OH group	0·06
Total	9·36

In both these examples non-polar van der Waals forces contribute a considerable proportion of the total heat of adsorption. This is particularly striking, since the ionic nature of the substrates would be expected to favour the formation of polar bonds.

The part played in dyeing processes by each of the forces listed above may now be considered. The first can operate only with protein and polyamide fibres which contain charged groups. The negligible adsorption of simple inorganic ionic substances such as sodium chloride shows that

such forces and also induction forces of the second type are very small and may be ignored. This must be attributed to the fact that there will be strong interaction between the ions and the highly polarisable water molecules, leading to heavy hydration, which will shield the ions from interaction with the fibre. The formation of a chemical bond occurs only in the acid dyeing of protein and polyamide fibres, where the hydrogen ion is combined with ionised carboxyl groups in the substrate. Thus polar and non-polar van der Waals forces must be the main factor in dye adsorption. It has been shown that the assumption that the former type are alone responsible for dye uptake is incapable of explaining certain dyeing phenomena. We shall now postulate that non-polar forces are the principal source of the affinity of dyes for fibres and see how well this assumption explains the facts.

Non-polar van der Waals forces (dispersion forces) are due to interaction between atoms of the adsorbent and the adsorbed molecules and are additive over all the atoms that are sufficiently close together. They are of short range, since the force varies approximately as the inverse sixth power of the distance between interacting atoms, whereas polar forces vary approximately as the inverse third power of this distance. Therefore, for maximum attraction, a close fit is necessary between large areas of the substrate and the adsorbed molecule. In an aqueous environment non-polar bonding will occur predominantly between hydrophobic surfaces, since the presence of polar groups, rendering one surface hydrophilic, will attract water molecules to the surface and prevent the necessary close approach. These are precisely the conditions for maximum bonding due to the forces postulated by Meggy [8], and in aqueous environment it is not possible to distinguish between the two effects. Therefore, in what follows, references to bonding by non-polar van der Waals forces will be taken as including the contribution from the recombination of water molecules by removal of hydrophobic surfaces.

On this hypothesis the affinity of a dye for a fibre will depend on—

(*a*) The hydrophobic–hydrophilic nature of the fibre

(*b*) The hydrophobic–hydrophilic nature of the dye

(*c*) The possibility of close packing between dye and surfaces in the fibre

(*d*) The accessibility of the fibre to dyes.

NATURE OF FIBRES

Adsorption of water by fibres is believed to occur in two ways—some of the water is strongly bound to polar groups in the fibre, and some is much less strongly attached to form so-called "capillary water". The amount of strongly bound water should give an indication of the extent to which the fibre is hydrophilic, while the amount of capillary water should be a guide in deciding to what extent the fibre will be accessible to different types of dye. The shape of moisture adsorption isotherms, and the results of attempts to analyse these into the two components mentioned above, lead to the belief that much of the bound water is adsorbed at lower humidities, and that the increase in absorption at humidities approaching saturation is largely due to capillary water. We shall therefore take the regain at 70% R.H. as an indication of the hydrophilic nature of the fibre, and that at 100% R.H., when compared with that at 70% R.H., as an indication of the relative accessibility of the fibre to dyes. These quantities are tabulated below (Table I) [11, 12] with the fibres arranged by this criterion in order of increasingly hydrophilic nature.

TABLE I [11, 12]

Fibre	Moisture Regain at 70% R.H. (%)	Moisture Regain at 100% R.H. (%)
Vinyl fibres ...	Negligible	
Terylene ...	0·4	0·6
Nylon ...	4·6	7
Cellulose acetate	7·6	8
Silk	11·0	33
Wool ...	16·0	33
Viscose rayon	16·0	33

The newer synthetic fibres of the vinyl type are extremely hydrophobic with negligible water uptake, as also is Terylene, which again has a very small moisture regain. Neither fibre has polar groups likely to combine with water by hydrogen bonding. The good tensile properties of Terylene are attributed to non-polar attraction between the benzene rings of the fibre [13]. This will lead to a close-packed structure which will be very resistant to swelling in water. Nylon is the next most hydrophobic fibre, in spite of its high content of polar amide groups. This must be attributed to the high degree of order and absence of bulky side-chains, which allow close packing and formation of interchain polar bonds, so that most of the polar groups are internally satisfied in this way. Cellulose acetate is almost as hydrophobic as nylon and very much more so than cellulose. This must be due to substitution of two-thirds of the hydroxyl (OH) groups in cellulose by acetoxyl (CH_3COO) groups. This fibre also shows a very small uptake of capillary water. In a recent thesis Rossett [14], who worked on a series of progressively acetylated celluloses, concluded that, at the degree of acetylation corresponding to the commercial product, interchain cohesion is due to non-polar van der Waals forces between glucose ring structures, which leads to a very close-packed structure.

The two natural protein fibres, silk and wool, are both more hydrophilic than the previous fibres, and wool is the more hydrophilic of the two. Both contain a large number of polar groups in the peptide linkages, and since the presence of side-chains prevents the close packing that occurs in nylon, fewer interchain bonds will be formed and consequently more polar groups will be available for water adsorption. Wool also contains a large number of polar side-chains, while the majority of the silk side-chains are non-polar, and the more

hydrophilic nature of wool may be attributable to this fact. Both fibres undergo a large swelling in water, so that they should be easily accessible to dye.

Cellulose is a relatively hydrophilic fibre with a large swelling in water. It is, however, doubtful whether water is adsorbed appreciably in the more crystalline regions, and recent work on cellulose sols[15] suggests that the micelles are hydrophobic. Cellulose sols were prepared from native and mercerised wood cellulose and viscose rayon by peptisation with distilled water after hydrolytic depolymerisation. The sols contained cellulose micelles whose crystalline structure had the same lattice as the crystalline regions of cellulose fibres, and whose dimensions agreed with estimates of the dimensions of such crystallites. The sols were coagulated by addition of small amounts of electrolyte, from which it was deduced that the micelles were hydrophobic. This may be due to internal bonding of the hydrophilic groups in the ordered crystallites, which will prevent their combination with water molecules. In the more disordered, amorphous regions, internal bonding will be less complete and the free groups will render these regions hydrophilic.

NATURE OF DYES

All dyes have a conjugated ring structure that produces colour by selective absorption of light in the visible region of the spectrum and is hydrophobic, with strong non-polar bond-forming capabilities. In disperse dyes and the oxidised form of vat dyes this structure predominates, and the dyes are consequently almost completely insoluble in water. In those classes of dyes which are water-soluble the hydrophobic nature of the ring structure is overcome by the introduction of solubilising groups, usually sulphonic acid groups.

Thus all dyes contain large hydrophobic surfaces which are capable of forming non-polar bonds if they can be sufficiently well fitted to hydrophobic regions in the fibre. Since all fibres are linear polymers, this fit would be most complete for linear dye molecules. Planarity will also be essential to obtain a close fit of the whole area of the dye molecule. Thus the observation that linearity and planarity are essential to substantivity of direct dyes is in agreement with the postulate that attachment to the fibre is due to non-polar forces. Since such forces are additive, increase in the size of the molecule and hence of the molecular weight will lead to an increase of binding energy. This explains the correlation between affinity and molecular weight, examples of which are quoted by Vickerstaff[16] in a recent review. One would expect that a small molecule would have adequate affinity for a hydrophobic fibre, while large molecules would be required for a hydrophilic fibre. This is in agreement with the fact that small, disperse dyes give full depths on Terylene, cellulose acetate, and nylon, while they will dye cellulosic fibres only to very pale colours. The relatively hydrophilic cellulose is dyed with direct or vat dyes which have larger molecules. The failure of the large dye molecules to dye the more hydrophobic fibres must be attributed to inaccessibility, the activation energy for penetration of large molecules into the close-packed fibre structure being prohibitively large.

The observed similarity between heats of formation of solid dye and the dye–fibre complex from dye in solution is explicable on the assumption that non-polar forces are the source of energy in each case. The additivity of such forces over the whole molecule means that the heat of combination per molecule of dye will be determined only by properties of the dye molecule, provided that the fit between dye and fibre is as close as that between dye molecules in the solid dye.

Further evidence of the importance of non-polar forces is provided by work[7] on the affinity of vat dyes of the anthraquinone and carbocyclic types. For series of related compounds a close correlation was found between the affinity of the dyes for cellulose and the extinction coefficient of the dyes in solution. The latter quantity will be a function of the conjugation structure of the dye, on which its non-polar bond-forming capabilities will depend. Thus the correlation with affinity illustrates the importance of these bonds in dye–fibre attachment.

DYEING MECHANISMS

We will now consider the dyeing of each type of fibre to see how well the hypothesis that non-polar bonding is the principal source of affinity explains differences in the dyes used for different fibres.

(a) *Vinyl Fibres*

These fibres are extremely hydrophobic, to such an extent that they have negligible moisture regain. They are not penetrated appreciably by water molecules, and dyeing from aqueous medium is consequently difficult. For consideration of dyeing properties they may be conveniently divided into polyacrylonitrile and polyvinyl chloride types.

(i) Polyacrylonitrile

A representative of this type of fibre is Orlon, which is most commonly dyed with disperse dyes. These small, hydrophobic molecules should have adequate affinity for the very hydrophobic fibre, and dyeing difficulties seem to be largely due to low rates of dyeing. Thus, while pale colours may be dyed at conventional dyeing temperatures below 100°C., it has been shown[17] that heavier dyeings are obtainable by pressure dyeing above 100°C. Alternatively, much deeper colours may be obtained at the lower temperatures by the use of swelling agents such as *m*-cresol and aniline.

(ii) Polyvinyl Chloride

An example of this type of fibre is Vinyon, which, again, is an extremely hydrophobic substance. The dyeing of this fibre has been reviewed by Woodruffe[18], who divides the dyeing methods into three classes—

I Dyes soluble in Vinyon

II Dyes soluble in assistant—assistant soluble in Vinyon

III Dyes having surface affinity for fibre due to polar structure.

The first method is applicable only to certain azoic dyes, whose components are coupled on the fibre to yield the coloured substance. All these substances are strongly hydrophobic, and their solubility in the substrate allows complete penetration. The third method is used only with basic dyes, which on application from an aqueous bath give heavy surface adsorption but no penetration into the fibre.

Method II may be used with dyes of the disperse, direct, and basic types by proper choice of assistant. The essential characteristics of the assistant used are that it shall be soluble in the fibre and act as a solvent for the dye. Its solubility in the fibre permits it to penetrate, while water will not, and it then provides a diffusing medium in the substrate for penetration of dye. Disperse dyes may be applied from aqueous dispersion with addition of an assistant, which may be either water-soluble (e.g. *iso*butyl methyl ketone) or water-insoluble (e.g. dibutyl phthalate). Alternatively, they may be dyed by the solvent-dyeing technique, in which the assistant acts as the dyebath with the disperse dye dissolved in it. This latter technique appears to be essential for dyeing with direct dyes, where the presence of more than about 5% of water in the dyebath inhibits dyeing, presumably because direct dyes are much more hydrophilic than disperse dyes. Basic dyes also may be dyed with assistants or by the solvent technique, when they penetrate the fibre, in contrast to their surface adsorption when dyed alone.

(*b*) *Terylene*

The experimental fact that this fibre is difficult to dye would be expected from the structure that has been outlined. The close-packed structure of benzene rings which suffers negligible swelling in water is analogous to that of graphite, in which sheets of covalently linked carbon atoms are bonded by non-polar van der Waals forces. Adsorption on graphite, other than on the exposed surfaces, is an activated phenomenon in which energy of activation is required to force apart the sheets of carbon atoms to allow adsorbed molecules to penetrate. It is likely that the dyeing of Terylene is similarly an activated process, and one important aspect of "carrier action" is to open up the fibre structure to allow dye to penetrate.

Carriers used are normally fairly small organic molecules containing aromatic rings (e.g. phenol, naphthalene, diphenyl). Their adsorption will be due to non-polar bonding of this ring to the benzene rings of the fibre, and their small size will permit penetration of the Terylene structure. In those carriers such as phenol which contain a polar group, non-polar forces must still be the main attractive force, although the hydroxyl group may form a weak polar bond with the ester linkage adjacent to the benzene ring. It is interesting that Peters and Sumner (quoted by Vickerstaff[19]) have found from direct comparison measurements with phenol and diphenyl that the uptake of the polar compound is much less than that of the non-polar compound. This indicates that the mechanism of combination is essentially non-polar, and that the polar group in the carrier, with much greater affinity for water than for the fibre, decreases the uptake of carrier.

Since the fibre is so hydrophobic, one would expect hydrophobic dyes of small size to have an adequate affinity for it, and disperse dyes having these characteristics are in fact used. Their small size is additionally advantageous in minimising penetration difficulties.

The picture of the dyeing mechanism, then, is of an initially close-packed fibre structure opened up by adsorption of carrier, whose affinity is sufficient to provide the activation energy to swell the fibre. The hydrophobic disperse dye has a high affinity for the very hydrophobic fibre and can penetrate to all parts after swelling. It will be attached mainly by non-polar forces to the aromatic rings of the fibre, in which position polar groups in the dye may also form weak polar bonds with the ester groups.

(*c*) *Cellulose Acetate*

This fibre also is hydrophobic and shows small swelling in water, but neither characteristic is so extreme as in Terylene. Residual hydroxyl groups will make the fibre slightly hydrophilic, and non-polar forces between the glucose rings will not be so strong as between the aromatic rings in Terylene, so that activation energies for penetration of dye molecules will be less. Because it is largely hydrophobic, this fibre also may be dyed with the small hydrophobic molecules of disperse dyes. Since the interchain cohesion is lower than in Terylene, the dyes can be used without carrier, their affinity providing sufficient activation energy to permit penetration. Carriers may be used to increase the rates of dyeing of disperse dyes (unpublished results obtained by Sumner), when their action must be similar to that on Terylene. Because of the residual hydroxyl groups in the fibre, polar groups in the carrier may be advantageous to bond to the hydroxyl groups with elimination of water to permit a closer fit of the hydrophobic surfaces. Phenol is particularly effective in swelling cellulose acetate, and substances of similar constitution to the fibre (e.g. esters) have been found to be effective as carriers.

Cellulose acetate fibres may also be dyed with water-soluble dyes of the Solacet type. These compounds have small molecules, so that penetration of the fibre is not difficult, and are solubilised by SO_3Na or SO_4Na groups. In the ionised form they may be attached to the fibre with their hydrophobic part bonded to the fibre and their charged groups in the hydrophilic environment provided by the residual hydroxyl groups in the substrate. It is also possible that they are not fully ionised in aqueous solution, and unionised dye, since it is more hydrophobic than the ionised form, will have a higher affinity for the fibre. This incomplete dissociation is suggested by

experiments in which dyes of this type were partitioned between water and organic solvent (e.g. amyl acetate), when substantial amounts of dye were found in the organic phase. It is notable that the solubilising group of these dyes is frequently very close to a basic group, which may modify their strengths as electrolytes.

(*d*) *Nylon*

Moisture regain figures for nylon are similar to those for cellulose acetate. The fibres have therefore similar hydrophobic–hydrophilic natures. It is significant that both fibres may be dyed with disperse dyes and Solacet dyes. The mechanism of dyeing with disperse dyes must be postulated as similar to that for cellulose acetate. Since nylon contains free amino and carboxyl groups, dyeing with Solacet dyes will be sensitive to pH, since, under acid conditions, the affinity of the hydrogen ion will contribute to the overall affinity.

The presence of amino and carboxyl groups makes it possible to dye nylon with acid dyes in a similar manner to the dyeing of wool. The free amino groups liberated by combination with acid are undoubtedly hydrated, and probably supply a suitable hydrophilic environment for the sulphonic acid groups in the dye. There are, however, significant differences between the two fibres. Nylon may be much more readily "overdyed"; i.e. the fibre will absorb dye at lower pH values in amounts far in excess of the number of free charged sites. This occurs to a much greater extent, and at higher pH values, than the analogous behaviour with wool. Nylon will adsorb level-dyeing acid dyes under neutral conditions much more readily than wool, and the affinity of such dyes is higher for nylon than for wool. Thus Metanil Yellow has an affinity for nylon of −18·5 kcal. per mole [20], while its affinity for wool is −11·4 kcal. per mole [2a]. These points of difference would be expected on the present hypothesis, since nylon is much more hydrophobic than wool. Consequently the hydrophobic part of the dye anion will have a greater affinity for nylon than for wool, and substantivity will be less dependent on the affinity of the hydrogen ion. However, the hydrophilic sulphonic acid group decreases the affinity of the dye anion, and dyes with more than one such group require very large affinities from the rest of the molecule to dye nylon under neutral conditions. Such dyes are to be found on the Carbolan range.

(*e*) *Wool and Silk*

These fibres are fairly hydrophilic, but they must still contain hydrophobic regions in the non-polar side-chains. Their high content of acid and basic groups makes them suitable for dyeing with acid dyes, as the number of such groups is well in excess of that necessary to give depths of colour normally required. Equilibrium studies of acid uptake as a function of pH have shown that adsorption of dye is generally similar to that of inorganic acids such as hydrochloric acid, with the difference that a given quantity of dye is adsorbed at higher pH values than the same quantity of hydrochloric acid. Adsorption of hydrogen ion by virtue of its affinity for carboxyl groups in the wool must be accompanied by adsorption of anions in equal quantity to maintain electrical neutrality. When chloride ion, with negligible affinity for the fibre, is adsorbed it will add nothing to the total affinity. But the dye anion has a large hydrophobic surface, which may be attached by non-polar forces to a hydrophobic surface in the wool, and the resulting decrease of free energy will contribute to the total affinity. The importance of this contribution is emphasised by the behaviour of Carbolan dyes, which have much higher affinities [21] than level-dyeing acid dyes. This must be due to the large increase in hydrophobic dye surface conferred by the long hydrocarbon chain. This will greatly enhance the non-polar bond-forming capabilities of the dye and so increase the total affinity. It should also be noted that Carbolan dyes have adequate affinity under neutral conditions, while corresponding levelling dyes do not readily dye neutral.

The effect of the free NH_3^+ groups produced by adsorption of hydrogen ions on to carboxyl groups is not certain. An interesting observation [22] is that wool dyed to fairly heavy depths with Carbolan dyes under acid conditions is hydrophobic, whereas uptake of the same dye from a neutral bath produces a dyeing which is hydrophilic. It seems, therefore, that the presence of free NH_3^+ groups under acid dyeing conditions produces a charged environment to which the solubilising group of the dye is attracted, while its hydrophobic part is attached to the fibre by non-polar bonding. In the absence of free NH_3^+ groups under neutral conditions such a charged environment does not exist and, while the hydrophobic portion of the dye is still attached by non-polar forces, the solubilising group remains attracted to the aqueous phase and so produces a hydrophilic dyeing.

(*f*) *Cellulose*

This fibre may be dyed with direct and vat dyes, which are similar in that both have large hydrophobic surfaces. Direct dyes are rendered water-soluble by their ionisable groups and they frequently contain a number of polar substituents. On the other hand, certain vat dyes contain no polar groups except two O^- groups in the reduced form of the dye. Thus in direct dyes the hydrophobic surface may be masked by hydrophilic groups while in some vat dyes it is not. Since both classes of dye have affinity for cellulose, it would seem that this affinity must again be due predominantly to non-polar van der Waals forces. Cellulose is a hydrophilic fibre, and one would therefore expect that large molecules would be necessary for adequate substantivity by this mechanism. Both types of dye fulfil this requirement, while the smaller disperse dyes will dye cellulose only to very pale colours. The large swelling which the fibres undergo in water permits adequate penetration of the dye molecules.

We have shown that the importance of linearity and planarity for substantive direct dyes is in agreement with the postulate of non-polar bonding. The presence of polar groups in direct dyes may

be essential to form bonds with the polar groups in the substrate, with elimination of bound water, to allow the necessary close approach of the non-polar bonding surfaces. With a hydrophilic fibre it may be necessary for the dye to have sufficient hydrophilic character to penetrate the water-swollen region of the fibre to achieve the close approach necessary for attachment by non-polar forces. Thus vat dyes in the unionised, insoluble state are not substantive, since in this hydrophobic form they are not able to pass through the hydrophilic cellulose environment and achieve this close approach. Solubilisation by reduction to form ionised groups will allow this penetration, when bonding will occur in a similar manner to that for direct dyes. In those vat dyes (e.g. pyranthrone) which have no polar bond-forming groups but high affinity for cellulose, the very large hydrophobic surfaces must have sufficient non-polar bond-forming powers to attach themselves in this way in spite of the presence of hydrated polar groups on the fibre.

Conclusions

The postulate that the principal contribution to the free energy of dyeing is from non-polar van der Waals forces, which occur in aqueous solution between hydrophobic surfaces of the dye and the fibre, explains the methods of dyeing different fibres. It predicts that small hydrophobic dye molecules will have adequate affinity for a hydrophobic fibre, while larger molecules will be required to dye a hydrophilic fibre. The additive and short-range nature of non-polar forces, with the consequent requirement of close fit of large areas of dye and substrate, explains the chemical criteria of linearity and planarity in substantive direct dyes and the general correlation between affinity and molecular weight in dyes of a particular class. Neither the increased affinity of Carbolan dyes due to a hydrocarbon chain nor the dyeing of Terylene can be interpreted in terms of polar forces, but both are readily explained on the assumption of non-polar bonds.

Formation of polar bonds is probably of importance only to eliminate water molecules bound to polar groups in the fibre, which would otherwise prevent the necessary close approach of dye and fibre surfaces. In the attachment of direct dyes to the hydrophilic regions of cellulose, formation of such polar bonds may be essential. The affinity of certain vat dyes having no polar groups may also be explained by non-polar forces if the very large area of hydrophobic surface in such molecules is adequate to bind to the fibre by this mechanism in spite of the presence of hydrated polar groups in the substrate.

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The Iodination of Wool

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When wool is iodinated with aqueous solutions of iodine in potassium iodide at different pH values and temperatures, reaction with the tyrosine side-chains is incomplete, even under the most favourable conditions. With alcoholic solutions of iodine, however, 96% of the tyrosine is converted into 3:5-di-iodotyrosine in 72 hr. at 22·2°C., and there is no combination with either histidine or tryptophan.

The fact that tyrosine can be iodinated exclusively and almost completely by such a simple procedure affords a useful means of discovering whether the lysine and tyrosine side-chains are cross-linked when wool is treated with formaldehyde. Since the amount of iodine which combines with the formaldehyde-treated wool, after loosely held formaldehyde has been removed with phosphoric acid, is the same as for untreated wool, it is concluded that the lysine and tyrosine side-chains are not cross-linked by formaldehyde. It has been shown also that the distribution of tyrosine along the length of single fibres can be determined by taking radiophotographs after the fibres have been iodinated with ^{131}I.

As a basis for a number of other investigations, including those which are discussed later in this paper, some method of converting all the tyrosine side-chains of wool into 3:5-di-iodotyrosine was required. No such method was available, in spite of earlier work on the action of iodine on wool by Haller[1], vom Hove[2], Haller and Holl[3], and Blackburn and Phillips[4]. The last-named authors treated wool with aqueous solutions of iodine in potassium iodide for one hour at room temperature and found that the iodine removed by the wool is of three kinds—absorbed iodine, combined iodine, and anionic iodine. It was suggested that the combined iodine represents substitution of iodine in the tyrosine side-chains to give 3:5-di-iodotyrosine side-chains, but the maximum amount of combined iodine recorded by Blackburn and Phillips, viz. 2·14%, is only about one-third of the amount to be expected if all the tyrosine side-chains are iodinated. Haller and Holl, however, obtained higher values for the amount of chemically combined iodine by a curious procedure. The wool was treated with an aqueous–alcoholic solution of iodine in potassium iodide for 24 hr., the volumetric proportions of alcohol and water being 1 : 4. After being washed, the wool was boiled with distilled water until no more iodine was removed, and the amount of iodine retained by the wool was estimated by the Carius method. The amount was found to be 6·5%, which is roughly what would be expected if the tyrosine were converted into 3:5-di-iodotyrosine, but the authors attribute combination to the formation of iodamine groups and regard substitution in tyrosine side-chains as taking place only to a trifling extent. It became clear, therefore, that a method for the complete and exclusive iodination of the tyrosine side-chains of wool could not be devised without further work on the action of aqueous and alcoholic solutions of iodine on wool. The development of such a method is the main subject of this paper, but two illustrations of its uses are included.

Experimental

PURIFICATION OF WOOL

After being rinsed in ether, the Lincoln shoulder wool used in the following experiments was extracted for 10 hr. each with alcohol and ether in a Soxhlet apparatus. The wool was then washed in running water for 24 hr., immersed in a large excess of dilute hydrochloric acid (pH 3) for 24 hr. to minimise the ash content, and finally washed with several changes of distilled water until the pH value of the solution in equilibrium with the wool was 5·4. At this stage the wool was removed, centrifuged, and stored in the humidity room for three weeks. After adhering vegetable matter had been removed from the conditioned wool with forceps, the fibres were cut into 5-mm. lengths, which were well mixed to ensure adequate sampling. All samples were weighed out in the humidity room, and the regain was determined by drying one sample from each set over phosphorus pentoxide *in vacuo* until constant weight was attained.

DETERMINATION OF TYROSINE CONTENT

Tyrosine was determined by the method of Block and Bolling[5]. Closely agreeing results were obtained in three distinct experiments, the amounts of tyrosine present in the Lincoln wool being found to be 4·79, 4·77, and 4·77%. Conversion of this amount of tyrosine into 3:5-di-iodotyrosine would give 6·70% of combined iodine (based on the dry weight of untreated wool).

I—Development of a Method for the Complete and Exclusive Iodination of Tyrosine

A. IODINATION WITH AQUEOUS SOLUTIONS OF IODINE

(i) *Influence of pH and Time of Treatment*

Four solutions of iodine were buffered to pH values of approx. 7, 8, 9, and 10 with Clarke and Lubs buffers[6]. The concentration of potassium iodide present was 0·2 M. and the amount of iodine was sufficient to produce a 0·1 N. solution in the absence of reaction with the buffering agents. A glass electrode was used for pH measurements, and the concentration of free iodine was determined by titration with 0·05 N. sodium thiosulphate solution. When the solutions were acidified, the hypoiodite present in alkaline solutions was converted into iodine, and titration gave the concentration of "potential free iodine".

Duplicate samples of conditioned, finely cut wool, each weighing 0·400 g. (0·338 g. dry weight), were inserted into drawn-out test-tubes (8 in. × 1 in.), and after addition of the iodinating solution (20 ml.) the tubes were sealed and clamped in a revolving shaker. The tubes were revolved

TABLE I

Free Iodine (N.)	Potential Free Iodine (N.)	Initial pH	Final pH	Time of Treatment (hr.)	Decrease in Free Iodine (% on dry wt. of wool)	Combined Iodine
0·100	0·100	6·85	6·43	4	23·7	1·82
0·100	0·100	7·92	6·57	4	23·5	2·58
0·096	0·097	8·94	8·58	4	21·4	3·16
0·069	0·095	10·15	9·09	4	19·1	3·22
0·100	0·100	6·85	6·04	8	26·1	1·98
0·100	0·100	7·92	6·30	8	26·2	2·78
0·096	0·097	8·94	8·10	8	24·9	3·89
0·069	0·095	10·15	8·78	8	22·0	3·95
0·100	0·100	6·85	5·47	16	28·1	2·14
0·100	0·100	7·92	5·72	16	28·6	3·01
0·096	0·097	8·94	7·32	16	27·7	4·46
0·069	0·095	10·15	8·21	16	25·7	4·49
0·100	0·100	6·85	4·71	32	30·3	2·23
0·100	0·100	7·92	5·02	32	31·1	3·20
0·096	0·097	8·94	6·36	32	31·4	4·74
0·069	0·095	10·15	7·13	32	29·1	4·81
0·100	0·100	6·85	4·31	64	32·0	2·33
0·100	0·100	7·92	4·68	64	33·2	3·45
0·096	0·097	8·94	6·47	64	33·7	4·90
0·069	0·095	10·15	6·47	64	31·3	4·95

(60 r.p.m.) for the required time in the dark at 22·2°C., and the solutions were then removed for determinations of pH and iodine content. Sodium thiosulphate solution (50 ml., 0·2 N.) was added to the wool and left for 24 hr. with occasional shaking, after which the solution was removed and the wool washed with distilled water. Each sample was then transferred to hydrochloric acid (200 ml., 0·01 N.) and left for 24 hr., before being immersed in distilled water, which was changed each day until no iodide could be detected. The amount of combined iodine in the wool was then determined in the following manner.

Each sample of wool was transferred to a 4-cm. nickel crucible, and after saturated sodium hydroxide solution (10 ml.) had been added, the mixture was left on the steam bath for 1 hr., by which time the wool had partly dissolved to give a yellow sludge. The product was heated gently with a Bunsen burner until all frothing had ceased, and then more strongly until a clear melt was obtained with small particles of carbon at the bottom. When cool, the mass was dissolved in hot water, and the solution filtered through a sintered glass crucible (No. 3), the filtrate and washings being made up to 100 ml. Samples (25 ml.) were pipetted into small conical flasks, sodium bisulphite solution (2 drops, 1%) was added, and the flasks were shaken. Methyl orange (1 drop) was added, followed by sulphuric acid (50%) until the solution was just acid, and then three drops in excess. The solution was warmed to 50°C., bromine water (3 ml.) was added, and after the mixture had been boiled for 1 min. it was cooled and treated with phenol (6 drops, 90%) to destroy any excess of bromine. A few crystals of potassium iodide were then added, and the liberated iodine was titrated with sodium thiosulphate (0·02 N.). Three aliquots were titrated in each case, and the average values for the amount of combined iodine are given in Table I.

(ii) *Influence of Temperature of Treatment*

As the maximum amount of combined iodine recorded in the preceding experiments is insufficient fully to iodinate all the tyrosine present in the wool, further experiments were carried out at higher temperatures. Each sample of wool was iodinated with the solution of iodine in potassium iodide at pH 6·85 for 8 hr., the sealed test-tubes being revolved in a water thermostat at the required temperature. The experiments were carried out in duplicate and the results are given in Table II.

TABLE II

Temp. (°C.)	Final pH	Decrease in Free Iodine (% on dry wt. of wool)	Combined Iodine
20	5·29	31·1	1·86
30	4·54	35·5	1·88
40	3·46	39·8	1·90
50	2·50	42·0	1·92

It will be noted that the decrease in the free iodine content of the solution is greater at 20°C. than in the preceding experiment at 22·2°C.; this is because the solutions were exposed to light, whereas the first experiments were carried out in the dark. The amount of combined iodine increases only slightly with increasing temperature of treatment, and different conditions of iodination are clearly needed if full iodination of the tyrosine is to be realised.

(iii) *Iodination in Presence of Concentrated Alkali*

According to Li [7], phenols are rapidly iodinated in concentrated alkali, but only if the phenol and the alkali are mixed first and the iodine is added afterwards. In an attempt to obtain more complete iodination of tyrosine, therefore, concentrated alkali was used in the following experiments. Each sample of wool (0·338 g. dry weight) was placed in a conical flask, and sodium hydroxide solution (20 ml., 0·123 N.) added at 22·2°C. After 1 min., iodine solution (2 ml., 0·4 N.) was added every 2 min. up to a total of 20 ml. The flask was shaken for a further 2 min. before the solution was removed, and the wool prepared as before for the determination of combined iodine. A second experiment was carried out in which water (40 ml.) was added to the wool before the sodium hydroxide solution, all other conditions being the same as before, while in a third experiment the conditions of the first were used, except that the iodine solution (1 ml., 0·4 N.) was added every 5 min. up to a total of 20 ml. All experiments were carried out in duplicate and the results are given in Table III.

TABLE III

Experiment	Combined Iodine (% on dry wt. of wool)
1	3·77, 3·73
2	2·44, 2·43
3	3·59, 3·66

(iv) *Iodination in Presence of Ammonia*

As the tyrosine of soluble proteins has been iodinated fully with an ammoniacal solution of iodine [8], where the iodinating agent may be nitrogen tri-iodide, the same procedure was applied to wool. Solutions were prepared by mixing solutions of iodine in potassium iodide with ammonia solution and a borate buffer to give different pH values. Each sample of wool (0·421 g. dry weight) was treated with the solution of iodine (20 ml.) in a sealed test-tube for 72 hr. at 22·2°C., the tube being revolved during the whole period of treatment in the dark. The experiments were carried out in duplicate and the results are given in Table IV.

(v) *Mode of Combination with Iodine*

In the preceding experiments, the greatest amount of combined iodine was obtained in the first series (Table I), and although the amount is less than that required for the complete conversion of tyrosine into 3:5-di-iodotyrosine, it may not all be combined with tyrosine. Other acids which may be iodinated are histidine, tryptophan, and proline. In order to discover whether they do react with iodine, the amino acids were treated with a solution of iodine in aqueous alcohol, and the reaction products examined by paper chromatography, iodination being carried out with such a solution in order to avoid the introduction of salts and eliminate the difficulties caused by their presence. Tyrosine (0·04 g.), histidine (0·2 g.), tryptophan (0·2 g.), and proline (0·2 g.) were dissolved in 50% alcohol (20 ml.) and to each was added 50% alcohol (20 ml.) saturated with iodine at 17°C. The solutions were well shaken, and left in a thermostat at 60°C. for 18 hr. before being run on Whatman No. 4 filter paper for 18 hr. at 17°C. Each spot contained 20 μg. of amino acid, and separate runs were made in butanol–acetic acid–water and phenol–water [9], in which solvents the iodine was taken to the solvent front and did not affect the R_f values of the amino acids. The chromatograms were then removed, dried, sprayed with a solution of ninhydrin in butanol, and heated for 3 min. at 80°C.

Iodinated tyrosine gave three spots, the strongest and weakest being identical with tyrosine and 3:5-di-iodotyrosine respectively. The spot of intermediate intensity, lying between the others, was found not to be due to thyroxine and was taken to be mono-iodotyrosine. Histidine, after iodination, gave very faint traces of two new spots, one with a slightly greater and one with a slightly smaller R_f value than histidine. Iodinated tryptophan gave the spot corresponding to tryptophan, with faint blurs in front and behind; and iodinated proline gave a single yellow spot, identical with that of proline.

When wool is iodinated, therefore, it seems clear that the main reaction is with tyrosine, though there may be some slight reaction with histidine and tryptophan. As, however, the conditions of iodination of the amino acids were different from those used in the first series of experiments with wool, a further experiment was carried out.

Duplicate samples of wool were iodinated in the dark, as before, for 64 hr. at different pH values and 22·2°C. Loosely held iodine was then removed with sodium thiosulphate, and the washed samples were used for estimations of combined iodine and tyrosine. Further samples of wool were iodinated under the same conditions and used for the isolation of histidine and tryptophan [5]. The histidine was found to be free from iodine, but small amounts were found in the tryptophan, as

TABLE IV

Initial Conen. Iodine (N.)	Initial Conen. Ammonia (N.)	Initial pH	Final pH	Decrease in Free Iodine (% on dry wt. of wool)	Combined Iodine (% on dry wt. of wool)
0·1	0·05	8·83	5·69	32·4	4·44
0·2	0·05	8·84	5·35	50·1	4·65
0·2	0·2	9·51	6·61	60·1	4·02
0·2	0·3	9·62	6·75	49·7	3·95

TABLE V

Initial pH	Tyrosine Content	Tyrosine iodinated (by difference)	Iodine equiv. to* Tyrosine iodinated	Combined Iodine	Iodine in Tryptophan
	(% on dry weight of untreated wool)				
Untreated	4·78	—	—	—	—
6·85	3·31	1·47	2·06	2·31	0·06
7·02	2·63	2·15	3·01	3·44	0·06
8·94	1·89	2·89	4·05	4·90	0·10
10·15	2·10	2·68	3·76	4·90	0·10

* Calculated on the assumption that the tyrosine side-chains are converted into 3:5-di-iodotyrosine

shown in Table V. Even when the amount of iodine combined with tryptophan has been taken into account, there is still a large discrepancy between the estimated amount of combined iodine and that calculated from the fall in tyrosine content on the assumption that the tyrosine is converted into 3:5-di-iodotyrosine. The discrepancy could, however, be accounted for if mono-iodotyrosine is estimated as tyrosine by the Block and Bolling procedure [5].

B. IODINATION WITH AN ALCOHOLIC SOLUTION OF IODINE

As it seemed to be impossible to iodinate the tyrosine side-chains of wool fully by means of aqueous solutions of iodine, attention was turned to alcoholic solutions. Absolute alcohol was saturated with iodine at room temperature (0·78 N.), left for 24 hr., and filtered, and the filtrate used as the iodinating agent. Each wool sample (0·421 g. dry wt.) was placed in a drawn-out test-tube and the iodine solution (50 ml.) added. The tube was then sealed and revolved in a thermostat at 22·2°C. for 72 hr. in the dark. After the wool had been removed and washed with absolute alcohol (50 ml.) for two periods of 1 hr., it was left in sodium thiosulphate solution (100 ml., 0·1 N.) for 18 hr. At the end of this time the wool was white and was transferred to distilled water (200 ml.), which was changed every 24 hr. until no iodide could be detected. Four such experiments were carried out, two samples of wool being used for determinations of combined iodine, and two for tyrosine estimations. The results are given in Table VI.

TABLE VI

Combined Iodine (% on wt. of wool)	Tyrosine iodinated* (%)	Decrease in Tyrosine Content (%)
6·48	96·7	96·0
6·42	95·8	95·2

* Calculated on the assumption that all the combined iodine is present as 3:5-di-iodotyrosine.

When histidine and tryptophan were isolated [5] from samples of wool which had been iodinated under the above conditions, they were found to be free from combined iodine. As will be indicated later in this paper, the fact that the observed decrease in tyrosine content of the wool is a little less than the calculated amount of tyrosine iodinated is due to the presence of tip wool in the sample. The values are, however, in such close agreement that it seems safe to conclude that about 96% of the tyrosine side-chains of wool can be converted into 3:5-di-iodotyrosine side-chains by iodination with alcoholic solutions of iodine at room temperature.

II—Some Applications of Iodination

As soon as a simple method of converting the tyrosine side-chains of wool into 3:5-di-iodotyrosine had been evolved, it became evident that the reaction had a number of interesting applications. Two of the simpler ones will now be described, and others will be discussed in later papers.

A. ACTION OF FORMALDEHYDE ON KERATIN

In accordance with the great weight of experimental evidence, the accepted view of the action of formaldehyde in hardening and strengthening protein fibres is that cross-linkages are formed between basic and amide side-chains. Alexander, Carter, and Johnson [10] have, however, suggested that when wool is treated with formaldehyde at 60°C. and pH 6–7, cross-linkages are formed between the phenyl groups of tyrosine and the ε-amino groups of lysine. In support of this view they draw attention to the fact that the irreversible weight increases obtained on treating wool with formaldehyde are of the order theoretically required for the postulated link, and the action of acid in breaking cross-linkages without liberation of formaldehyde is attributed to its retention as hydroxymethyltyrosine. More convincing evidence for the cross-linking of tyrosine and lysine side-chains in untreated wool would be the failure of formaldehyde to cross-link iodinated wool, and the action of formaldehyde on untreated and iodinated fibres was, therefore, examined.

Fibres of Lincoln shoulder wool (0·02 g.) were treated with formaldehyde solution (50 ml., 0·1 M.) for 30 min. at pH 6·3 and 60°C. and then washed in distilled water for 48 hr. Other fibres were iodinated with the alcoholic solution of iodine as described in § I(B), and half of the iodinated fibres were then treated with formaldehyde under the same conditions as the untreated fibres. It was expected that the supercontraction of the non-iodinated formaldehyde-treated fibres in a boiling 5% solution of sodium bisulphite would be less than 6%, in accordance with the results of

TABLE VII

Treatment	Supercontraction (%)	Supercontraction (%) after Treatment and heating with Phosphoric Acid
Untreated	34·8 ± 0·3	29·7 ± 0·5
Iodination	31·5 ± 0·6	40·0 ± 0·4
HCHO (60°C., pH 6·3) ...	28·0 ± 0·3	38·4 ± 0·2
Iodination and HCHO (60°C., pH 6·3) ...	36·8 ± 0·9	29·1 ± 1·2
HCHO (60°C., pH 1·2) ...	26·7 ± 0·2	37·4 ± 0·3
HCHO (reflux, pH 1·2) ...	1·9 ± 0·1	33·2 ± 0·4

Alexander *et al.*[10], and that the supercontraction of the iodinated formaldehyde-treated fibres would be very much greater because the formaldehyde could not form cross-linkages between 3:5-diiodotyrosine and lysine side-chains. The supercontraction of five fibres from each type of wool was determined under the usual conditions[11] and the average results are given in Table VII.

Formaldehyde-treated fibres, like iodinated fibres, supercontract only a little less than untreated fibres. This result is in sharp conflict with the observations of Alexander *et al.*[10], but agrees well with those of Stoves[12]. It is interesting that the supercontraction of fibres which were treated with formaldehyde after iodination should be greater than that of fibres treated with formaldehyde alone, but this result cannot be taken as evidence in support of the claim that the tyrosine and lysine side-chains of untreated wool can be cross-linked with formaldehyde, because the opposite result is obtained when the fibres are first freed from loosely bound formaldehyde with phosphoric acid. The results given in the last column of Table VII were obtained with fibres which had been immersed in phosphoric acid (100 ml., 0·3 M.) and heated on a hot-plate for 2 hr., during which time the volume was reduced to 10 ml. Before the supercontraction was measured, the fibres were washed in distilled water for 24 hr. In an attempt to obtain improved cross-linking with formaldehyde and, therefore, more conclusive results, more fibres (0·02 g.) were treated with formaldehyde solution (50 ml., 0·1 M.) for 30 min. at pH 1·2 and 60°C., but the supercontraction in presence and absence of loosely bound formaldehyde is only a little less than that of fibres treated at pH 6·3. Finally, fibres were boiled under reflux with formaldehyde solution (0·67 M.) for 1 hr. at pH 1·2, as recommended by Stoves, and then washed in distilled water for 48 hr. Considerable cross-linking was thus obtained, the supercontraction of the fibres being very much less than that of untreated fibres, as shown in Table VII. After being heated with phosphoric acid, however, the fibres are capable of supercontracting more than corresponding untreated fibres, and it seems probable that the cross-linkages are broken by the acid. Vulnerability to acid would be expected if tyrosine and lysine are cross-linked by formaldehyde at pH 1·2, but some more conclusive evidence for their formation is clearly necessary, especially in view of the small degree of cross-linking which is obtained under Alexander's conditions at pH 6·3. It seemed likely that such evidence might be obtained by iodinating formaldehyde-treated fibres, after removal of loosely bound formaldehyde, and determining the amount of combined iodine. According to Alexander, formaldehyde is retained as hydroxymethyltyrosine when the linkages between tyrosine and lysine are broken by acid, and the amount of iodine which combines with the treated wool should, therefore, be less than with the untreated wool.

Accordingly, wool samples (0·421 g. dry weight) were treated with formaldehyde under the same conditions as before, i.e. either with formaldehyde solution (50 ml., 0·1 M.) for 30 min. at pH 6·3 and 60°C., or with formaldehyde solution (50 ml., 0·67 M.) under reflux for 1 hr. at pH 1·2. To remove loosely held formaldehyde, each sample was transferred to phosphoric acid (100 ml., 0·3 M.), which was then placed on a hot-plate and evaporated down to 10 ml. over a period of 2 hr. For reference purposes, samples of untreated wool were treated with phosphoric acid in the same way. All the wools were then iodinated under the conditions described in § I(B), and the amounts of combined iodine determined. The results are given in Table VIII.

It is clear that there is no foundation for Alexander's suggestion that the lysine and tyrosine side-chains are cross-linked when wool is treated with formaldehyde at pH 6·3 and 60°C. Even under Stoves' conditions (pH 1·2, reflux) there is little, if any, reaction with tyrosine. It should be noted that the amount of combined iodine is expressed as a percentage of the dry weight of the untreated wool, and the low value is due, no doubt, to dissolution of a small amount of protein during the process of refluxing at pH 1·2.

B. DISTRIBUTION OF TYROSINE ALONG LENGTH OF WOOL FIBRE

It is well known that the sulphur content of wool varies along the length of the staple, in accordance with the nutrition available at the time of growth[13], though the sulphur content of the tips of the staples may be abnormally low in relation to nutrition because of loss of sulphur during exposure to light and air. How the content of other amino acids varies along the length of the

TABLE VIII

Treatment	Combined Iodine (% on wt. of wool)	Tyrosine iodinated (%)*
H_3PO_4	6·36	94·9
HCHO (60°C., pH 6·3) and H_3PO_4 ...	6·32	94·3
HCHO (reflux, pH 1·2) and H_3PO_4 ...	6·16	91·9

* Calculated on the assumption that all the combined iodine is present as 3:5-di-iodotyrosine

staple has not, so far, been studied, but it is clear from first principles that if the sulphur (cystine) content increases with increasing nutrition, the content of some other amino acids must diminish. With these considerations in mind, it was decided to compare the variations in sulphur and tyrosine contents along the length of staples of Lincoln shoulder wool.

A number of staples were divided into four sections, viz. root, tip, and two intermediate sections. Each section was purified separately in the manner already described, and the sulphur [14] and tyrosine contents were then determined on duplicate samples. Other samples were iodinated (§ I(B)) and the combined iodine was determined. The results are given in Table IX.

Over the first three sections, the fall in sulphur content is matched by a fall in tyrosine content, but the relationship breaks down with the tip wool owing to the effect of exposure on both the sulphur content and the tyrosine content. As would be expected, because of the absorption of ultraviolet radiation by the tyrosine side-chains, peptide bonds associated with tyrosine are specially vulnerable, and the tip wool is, therefore, deficient in tyrosine. Despite this deficiency, however, the amount of iodine which combines with the tip wool is almost the same as for section 3, and one effect of exposure to light and air must be to create new iodine-combining groups. One possibility is combined α-aminoacrylic acid, derived from cystine.

It is interesting that the tyrosine content of the Lincoln wool should vary along the length of the staple in the same general manner as the sulphur content, apart from the peculiarities of the exposed tip wool, and there must now be even greater interest in the effect of nutrition on the proportions of other amino acids besides cystine and tyrosine. Further, since the different fibres within a single staple of wool are now known to show remarkable variations in plasticity [15], which appear to be associated with variations in sulphur content and crystallinity [16], there is an urgent need for methods of determining the proportions of different amino acids in single fibres. Particular interest is associated with tyrosine in relation to variations in plasticity, because it plays an important part in promoting main chain disorder in the case of silk, and it seemed likely that a satisfactory micro method of estimating the tyrosine content of single fibres might be based on iodination with radio-iodine (^{131}I). The possibility was examined by studying the variations in tyrosine content along the length of single Lincoln fibres and comparing the results with those already available for staples of the same wool (Table IX).

A solution (0·58 ml.) of ^{131}I (10 millicuries) in the form of iodide, supplied by the Atomic Energy Research Establishment, was transferred to a small centrifuge tube and made up to 2 ml. with distilled water. A saturated solution of potassium iodate (0·5 ml.), containing concentrated sulphuric acid (10 ml./litre), was then added, and gave a faint yellow coloration. There was not enough iodide present to give a precipitate of iodine, and a solution of potassium iodide (5 ml., 0·1 N.) was added so that the precipitated iodine would act as a carrier for the ^{131}I. Potassium iodate solution was then added to complete the precipitation of iodine, and the product was centrifuged before the supernatant liquid was removed. The iodine was finally washed with distilled water (5 ml.) in the centrifuge tube, the wash waters being again removed after centrifuging, and, after adhering water had been removed by touching the iodine with filter paper, the iodine (0·137 g.) was dissolved in absolute alcohol (1·5 ml.).

Twenty Lincoln wool fibres (0·0247 g. dry wt.), with lengths of the order of 20–30 cm., were taken from a purified staple of shoulder wool and immersed in the solution of iodine in a 2-ml. glass tube. The tube was sealed, placed in a large test-tube in a thermostat at 25°C., and left for 72 hr. with occasional agitation. After the solution of iodine had been removed, the fibres were washed 20 times with absolute alcohol, and the solution and washings were made up to 250 ml.

TABLE IX

Section	Sulphur	Tyrosine	Combined Iodine	Tyrosine iodinated (%)*
	(% on dry wt. of wool)			
1 (Root)	3·77	4·92	6·65	96·3
2	3·68	4·84	6·44	94·9
3	3·34	4·63	6·06	93·3
4 (Tip)	3·36	4·56	6·07	94·9

* Calculated on the assumption that all the combined iodine is present as 3:5-di-iodotyrosine

TABLE X

Average Values of $1000/d^2\psi$

Fibre 1		Fibre 2	
5-mm. Lengths	25-mm. Lengths	5-mm. Lengths	25-mm. Lengths
3·21 Root		2·83 Root	
3·08		2·67	
3·12	3·13	2·80	2·74
3·20		2·74	
3·06		2·64	
3·10		2·80	
3·05		2·72	
3·04	3·05	2·67	2·75
—		2·76	
3·00		2·80	
3·04		2·70	
2·97		2·66	
3·00	2·98	2·65	2·64
2·97		2·62	
2·92		2·56	
3·02		2·76	
3·10		2·65	
2·98	3·01	2·62	2·61
2·99		2·61	
2·97		2·39	
3·02		2·56	
3·03		2·60	
2·98	3·02	2·57	2·58
3·08		2·60	
3·00		2·58	
3·06		2·56	
2·97		2·64	
3·03	(3·02)	2·62	2·64
3·02 Tip		2·66	
		2·70 Tip	

with absolute alcohol. This solution, and the aqueous solution from which the iodine had been obtained (made up to 250 ml.), were compared for radioactivity by means of a Geiger counter. It was found that nearly all (97%) of the ^{131}I was present in the alcoholic solution. The fibres were next placed in sodium thiosulphate solution (50 ml., 0·1 N.) for 48 hr., and finally in changes of distilled water (50 ml.) until the water, after 24 hr. contact with the wool, contained no ^{131}I. Finally, the fibres were dried over phosphorus pentoxide *in vacuo*.

After several methods had been tried, it was decided that the distribution of ^{131}I along the length of the fibre was best determined by taking a radiophotograph. For this purpose, small lengths of radio-iodinated fibre were tied round the root and tip ends of another radio-iodinated fibre to act as reference points. The fibre was then mounted on a glass frame under low tension and placed between Ilford Process plates. Satisfactory results were obtained with an exposure of 25–30 min., and, after development, the negative was placed in a sensitive photometer which gives a direct reading for the intensity of transmitted light. Readings were taken along the radiograph of the fibre at intervals of 1 mm. from the root reference point, and the diameter of the fibre was measured at the same points. If d is the diameter of the fibre and ψ is the photometer-reading for transmitted light, the blackening of the film (which is proportional to the amount of ^{131}I) per unit volume of the fibre is proportional to $1/(d^2\psi)$. Closely agreeing results were obtained with the two negatives for each fibre, and the average values over successive 5-mm. and 25-mm. lengths of two fibres are given in Table X.

In agreement with the results of Table IX, the average values of $1000/(d^2\psi)$ show that the tyrosine content of the fibres is greatest at the root and diminishes towards the tip. In addition, the amount of combined iodine is greater at the tip than in the middle of the fibre, in accordance with the results of gravimetric determinations. It may be concluded, therefore, that the distribution of tyrosine along the undamaged length of single wool fibres may be determined by iodination with ^{131}I and the preparation of radiophotographs. The results of Table X make it clear that there are differences in the tyrosine content of corresponding portions of fibres from the same fleece, and these differences, as well as differences in sulphur content, are worthy of further investigation in relation to the variations in plasticity among the fibres of a single staple.

* * *

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TEXTILE CHEMISTRY LABORATORY
DEPARTMENT OF TEXTILE INDUSTRIES
THE UNIVERSITY
LEEDS 2

(*Received 11th March 1955*)

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Notes

Election of Fellows and Associates

At the meeting of Council held on 14th September 1955, the following were elected either Fellows or Associates of the Society.

FELLOWS

John Austin Eastwood
(*Sydney, Australia; Dyehouse Manager, Australian Woollen Mills Pty. Ltd.*)

Charles William Green
(*Bruntcliffe, near Leeds; Technical Dyer, William Smith & Sons Ltd.*)

Philip Gordon Noble
(*Dumfries, Scotland; in charge of the Technical Service Section, Textile Development Department, Ardil Fibre Factory, Imperial Chemical Industries Ltd.*)

ASSOCIATES

Clifford Beaumont
(*Heywood, Lancashire; Research Chemist, Courtaulds Ltd., Droylsden*)

Noel Bradbury
(*Stalybridge, Cheshire; Printing Colourist, John Walton of Glossop Ltd.*)

James Harry France Hilton
(*Ashton-under-Lyne; Superintendent, Standardisation Laboratories, The Clayton Aniline Co. Ltd.*)

Samuel Whitbread Leech
(*Stockport; Dyehouse Manager, J. J. Hadfield Ltd.*)

Derek Reynolds
(*Bradford; Yarn Dyer, Holybrook Dyeing Co. Ltd.*)

William Russell
(*Coventry; Dyeing Technician, Courtaulds Ltd.*)

Jan Zdzislaw Sadowski
(*Manchester; Assistant Research Chemist, Courtaulds Ltd., Droylsden*)

Stuart Sidney Smith
(*Halifax; Apprentice Dyer, Joseph Gee & Sons Ltd.*)

James Watson
(*Troon, Scotland; in charge of dyehouse, The Hosiery Manufacturing Co. Ltd.*)

The following candidates have passed in Papers A and B at the Associateship Examination held in June 1955—

Jozef Biedawski
James Hawksworth
Ludomir Stanislaw Laskowski
John Lunt
Zygmunt Macheta
Fred Peach
Edmund Przetakiewicz
John Bennett Swanston

Meetings of Council and Committees

August

Council—No meeting
Publications—16th
Colour Index Editorial Panel—9th and 19th

Death

We regret to report the loss by death of Mr. Arthur Clayton, of Carlisle.

Fastness Tests Report

Council has placed on record its sincere gratitude to the Fastness Tests Co-ordinating Committee for its work culminating in the *Third Report* (J.S.D.C., **71**, 283–342 (June 1955)). The period intervening since publication of the second edition of the *Second Report* in 1950 has been occupied by vigorous activity on the part of the Subcommittees dealing with various individual tests, which has required much laboratory work by members. The Co-ordinating Committee has been largely occupied with the co-ordination of the Society's tests with those of other countries, notably the U.S.A. (A.A.T.C.C.) and the Continental countries (E.C.E.). Much time and energy have been spent, especially by the Chairman and the Honorary Secretary of the F.T.C.C., in attending international conferences, many held under the auspices of the International Organisation for Standardisation, at which detailed questions of testing have been thrashed out. The extent to which international agreement has been achieved may be regarded as a tribute to the efforts of the Committee, and

publication of the *Third Report* (shortly to be reprinted under the title *Standard Methods for the Assessment of the Colour Fastness of Textiles*) is an event of some importance both for the Society and in the international field.

The Society is deeply indebted to all members of the Co-ordinating Committee and of Sub-committees (whose names are listed in the July issue of the *Journal*) for devoting so much time and labour to this work, and is especially indebted to their employers—private firms and other organisations—for providing laboratory facilities and granting the time necessary for committee meetings and other activities. The practical goodwill to the Society demonstrated by the industry in allowing members to spend so much of their working time on the Society's business does not figure in the annual accounts, but it represents a very large "invisible asset".

Chair of Textile Chemistry University of Manchester

Dr. Raymond Harry Peters has been appointed the first Professor of Textile Chemistry and Director of the Laboratories in the Faculty of Technology (Manchester College of Technology). Dr. Peters graduated in chemistry at King's College, University of London, in 1939, and three years later obtained the degree of Ph.D. in organic chemistry. In 1949 he gained the B.Sc. degree of the University of Manchester with Honours in mathematics. Since 1952 he has been head of the dyeing research section at ICI Dyestuffs Division, Blackley, Manchester, and he was awarded the Research Medal of the Worshipful Company of Dyers for 1952–1953.

National Diplomas in Technology

It was announced in the House of Commons on 14th July 1955 by the Minister of Education, Sir David Eccles, that a National Council of Diplomas in Technology in Technical Colleges is to be established and that Lord Hives, chairman and managing director of Rolls-Royce Ltd., has been appointed chairman of the Council. Awards will be available to students in technical colleges in Britain who successfully complete technological courses approved by the Council. It is intended that the diploma award shall be comparable in standard with a first degree of a university. The question of a post-graduate award is to be considered later.

Manchester College of Technology Practical Classes in the Dyehouse

With the completion of the newly built and equipped dyehouse, the Department of Textile Chemistry is able to recommence realistic practical instruction in modern methods of textile processing. The Department is prepared to accept special students in its regular classes or to arrange special classes if the demand warrants it.

Nederlandse Vereniging voor Textielchemie

On 8th October 1955 the N.V.T.C. will hold an all-day meeting in Helmond for members and guests. Lectures will be given on "trouble shooting" and automatisation in textile production, and there will be a reception by the Mayor of Helmond. Further information is obtainable from the local organising committee, c/o Koninklijke Textielfabrieken J. A. Raymakers & Co. N.V., Helmond, Holland.

Society of British Printing Ink Manufacturers

The British Printing Ink Federation and the British Printing Ink Association are to amalgamate on 1st January 1956, when the new body will become known as the Society of British Printing Ink Manufacturers.

C. O. C.

OBITUARY NOTICE

George Herbert Frank

We learn with regret of the death of Mr. Frank early this year. He was active at meetings of the West Riding Section of the Society before the 1914–1918 War, and for a number of years was a member of the Publications Committee and also the West Riding Section Committee.

He was born at Kirbymoorside, Yorkshire, in 1884, and educated at Pickering Grammar School. He studied dye chemistry at the University of Leeds under the late Professor Green, taking his B.Sc.(Honours) in 1907, and later his M.Sc.; subsequently he became a Fellow of the Royal Institute of Chemistry. After graduating, he joined the teaching staff of the Department of Tinctorial Chemistry at Leeds University, and was a popular member of the staff and undoubtedly a brilliant teacher. The departure of G. H. Frank for industry was a very great loss to the teaching world, as he had a gift of handing on his knowledge to students, and a great sympathy for their approach to the work. During his career as a student himself, he was interested in the athletic life of the University, and was the winner of the Northern Universities mile for Leeds University.

During the 1914–1918 War, he left Leeds University and joined the research team which was being built up by British Dyes Ltd., subsequently staying with that organisation when it was enlarged to become the British Dyestuff Corporation Ltd.

In 1924 he joined Hickson & Partners Ltd. at Castleford, specialists in the manufacture of dye intermediates. He was at first works manager, and subsequently became a director.

In the last War, although he had retired from active work in the chemical industry, he again volunteered to assist the national effort, and worked for the Ministry of Supply on various of their activities.

In 1950, drawing on his former experience of the manufacture of dye intermediates, he wrote *The Manufacture of Intermediates and Dyes* (London: Constable & Co. Ltd.), which was reviewed in this *Journal* (**67**, 195 (1951)).

In 1920 he married Miss Frances Woodhead, of Huddersfield, who together with two sons survives him.

He retired to Truro in Cornwall after the last War, and his death occurred there.

B. HICKSON

New Books and Publications

A Handbook of Textile Dyeing and Printing

By A. J. Hall. Pp. vii + 202. London: The National Trade Press Ltd. 1955. Price, 21s. 0d.

On the basis of his wide experience, Mr. Hall has written a number of successful books on the chemistry of fibres and the wet processing of textile materials. His latest book, written in his usual easy style, is likely to prove very popular among textile students. It will also serve as an introduction for students of dyeing, whilst the non-chemical parts should be readily intelligible to those concerned with the sale of textiles.

The introductory chapter is followed by a brief but well illustrated chapter on textile fibres. Then follows a chapter on dyes. In this chapter, and to some extent in that on dye absorption, the author is faced with the problem of dealing with chemical topics in largely non-chemical language. He has, almost inevitably, been only partly successful, and over-simplification has resulted in some misleading statements. Thus, on p. 49, the Glauber's salt addition to an acid dyebath is said to enhance the solubility of the dye, whilst the purpose of the acid is to counteract the restraining effect of the Glauber's salt! In general, the author is less happy when dealing with wool dyeing than when discussing the dyeing of cellulosic or synthetic fibres. On p. 52 it is not clear whether he is referring to acid-dyeing or neutral-dyeing pre-metallised dyes, or to copper-containing direct dyes. Among errors noted were the statements that acid dyes are suitable for dyeing Terylene (p. 49), that toluene-*p*-sulphonyl chloride is still used for preparing immunised cotton (p. 93), and that *butyl*benzenesulphonate is a detergent (p. 180).

About half the book consists of two long chapters dealing with the mechanical side of dyeing and printing. These chapters, especially that devoted to textile printing, are very good and well illustrated: they form the most valuable part of the book. The two final chapters deal with textile auxiliaries and with fastness testing.

It is to be hoped that, before the second edition is published, the author will endeavour to remove the blemishes from this book, which otherwise is highly commendable.

C. L. BIRD

Chemische Technologie und Praxis der Färberei

Band I

Die Farbstoffe und das Färben der Wolle

By G. Nitschke. Pp. 256. Berlin: VEB Verlag Technik. 1954. Price, DM 20.00.

In a foreword to the series, the author outlines his aim as being to collate in concise form the available knowledge of dyes and fibres, relating these at the same time to practice. As regards this first book on wool dyeing, he has succeeded in producing an orderly work with economy of space. Each class of dye normally applied to wool is considered from a number of standpoints, giving, as far as is possible, a regularity of treatment to each. Thus, the acid, metal-complex, mordant, and vat dyes are reviewed with respect to their constitution, mechanisms of dyeing, specific properties, techniques of application, fastnesses, and possible faults in processing. The newer developments, such as dyeing at reduced temperatures, the Steverlynck system, and the application of anthraquinonoid vat dyes to wool, are included in the scheme, as are new dyes of the weak-acid-dyeing premetallised type.

A well illustrated section on the machinery used in wool dyeing then follows, dealing in turn with the handling of wool in loose, top, yarn, piece, hat, and felt forms. Continuous dyeing processes are considered, and this section also includes a discussion of the common faults encountered in yarn and piece dyeing. The influence of the state of the fibre on the dyeing process, for example the effects of residual oil and prior light damage, and conversely the effect of the dyeing process on the fibre itself, are also reviewed. The final section of the book covers the use of auxiliaries in wool dyeing, including detergents and levelling, protecting, and softening agents. The stripping and the reserving of wool conclude the section.

As a whole, the book is rather similar to Bird's *The Theory and Practice of Wool Dyeing*, but its size is unnecessarily increased by the number and length of light fastness tables included. It should find a ready sale on the Continent and in South America, but in this country its popularity may be affected by the existence of a parallel, competent work and by the limitation of the products discussed to those of German and Swiss origin. For those

sufficiently interested, language and style should present little difficulty, since the book is singularly free from involved box sentences. It would be conventional for a reviewer, at a time when the critic is assuming more importance than the creator, to fault the details of this book, but in this case only the general impression leaves something to be desired. Technical authors obviously do not write with one eye on possible film rights, but this fact should not deter them from attempting a new slant.

In striving for order in knowledge, without originality a textbook of this nature can acquire the air of having been produced mechanically from abstracts and trade pamphlets. The present work appears to have been drably assembled rather than written on a colourful subject.

S. R. COCKETT

Die Polyamide

By H. Hopff, A. Müller, and F. Wenger. Pp. xii + 423. Berlin: Springer-Verlag. 1954. Price, DM 43.20.

This book deals with polyamides, including polyurethans, from the viewpoint of the manufacturer rather than that of the user. It is divided into three sections, covering the manufacture of polyamides (140 pp.), polyamides as raw materials in the plastics industry (124 pp.), and polyamides as textile materials (150 pp.).

The book is based on German experience in this field; consequently the polymers from caprolactam (Perlon) figure prominently. Although the authors discuss a number of routes for the production of caprolactam and other polyamide intermediates, including two routes from acetylene, it seems that the preparation from phenol is still the only economic method. A description is given of the preparation of the diisocyanates, the intermediates for the preparation of the polyurethans. Polyurethans are of little interest in the textile field, but they have considerable possibilities as plastics, for surface coatings, and for certain rubberlike materials.

The section dealing with polyamides as plastics covers the mechanical, electrical, and thermal properties; also their behaviour towards water, acids, alkalis, and solvents. Some of the information is surprising; for instance, copolymers of 6,6-nylon and caprolactam, in the ratio 1 : 1, melt in boiling water.

Textile fibres are by far the most important use of polyamides. In addition to the orthodox method of spinning from chips, the authors describe two methods, developed in Germany, which might have advantages when the standards of uniformity are less stringent, as when producing staple fibre. The first is the "band spinning" process. In this the polymer is first produced in the form of a ribbon of rather closely controlled dimensions. This ribbon is forced by means of serrated rollers into a slot in a heated metal block. The ribbon acts as a piston, and forces the molten polymer through a spinneret in the lower part of the block. The second process is the "V.K." (*vereinfachtes kontinuierliches Verfahren* = simplified continuous process). In this process, which can be applied only to caprolactam, the polymer is not isolated at an intermediate stage. Since the polymerisation of caprolactam does not involve the elimination of water, the reaction can be carried out under atmospheric pressure. Molten caprolactam, with the addition of a catalyst, is fed into the upper end of a heated tube. As it passes downwards it polymerises, and molten polymer is taken from the bottom of the tube directly to the spinnerets.

The book contains a full account of the various processes through which the fibre must pass after leaving the spinneret, including conditioning, winding, drawing, twisting, and sizing. For Perlon fibres a washing process is necessary to remove residual monomer. There is a description of methods for the production of staple fibre, including crimped fibres. The production of bristles is also included.

The section on the dyeing of polyamides covers 14 pages, and does not include anything of note that is not already available in English textbooks, or in publications in English journals.

The book is well printed and indexed, with numerous figures and tables, and 85 pages of patents abstracts. To the worker in this field who has a sufficient knowledge of German it should be a useful reference book.

A. B. MEGGY

Plastics Progress 1953

Papers and Discussions at the British Plastics Convention 1953

Edited by P. Morgan. Pp. vii + 439. London: Iliffe & Sons Ltd. 1953. Price, 50s. 0d.

New developments in the plastics industry are covered by papers on the use of polyamides and polyesters in moulded and extruded forms, polymers which in fibre form are better known under the names of nylon and Terylene. Glass-fibre-reinforced polyester–styrene resin systems are prominent in the papers. The most interesting contribution from the point of view of the textile industry is that on flameproof conveyor belting, now extensively employed in coal mines in this country. This type of belting involves the combined use of fabrics with polyvinyl chloride–plasticiser systems.

A. HILL

The Technology of Solvents and Plasticisers

By A. K. Doolittle. Pp. xv + 1056. New York: John Wiley & Sons Inc. 1954. Price, 148s. 0d.

This is an excellent book covering both the theory and the applications of solvents and plasticisers. One chapter is devoted to solvents for textile fibres and covers the solvent systems used in the dry spinning processes for cellulose acetate, acrylonitrile polymer and copolymers, and vinyl copolymers. This chapter also covers briefly the use of solvents in textile processing. The trends towards using polymer systems in textile finishes, including printing, and the enormous expansion in the use of polymer–plasticiser coatings on textile fibres and fabrics, the latter being mainly concerned with plasticised polyvinyl

chloride, make this book of value to the textile industry in its wider sense, in addition to those engaged directly in the more conventional plastics and surface-coating industries. A. HILL

The Physics of Particle Size Analysis

A Conference arranged by The Institute of Physics and held in the University of Nottingham on 6–9th April 1954

British Journal of Applied Physics Supplement No. 3. Pp. vi + 218. London: The Institute of Physics. 1954. Price, 35s. 0d.

Dust, in its widest connotation, increasingly appears either as an adjunct to, or as a by-product of, modern civilisation. It enters our lives literally from the cradle to the grave, since not only does it nourish and comfort the newly born, but also it is a killer in its own right, by way of pneumoconiosis and silicosis. It is the mainstay of the modern industrial architect, yet it fouls the cities that he builds. It protects man's crops, prints his newspapers, and beautifies his wife. The painter, the potter, and even the policeman, all find uses for dust. It is not surprising, therefore, that there has arisen an urge to know more about dust in all its forms, and particularly about the physical behaviour of the systems formed when dust is suspended in some fluid medium, whether liquid or gas. Such behaviour depends to a marked extent not only on the average particle size of the dust concerned, and its concentration in the suspending medium, but also on the particle size distribution, and in some cases on the particle shape.

The precise determination of these properties of a dust (or droplet) suspension has proved to be a matter of considerable difficulty. Naturally enough, the first instrument employed for the purpose was the optical microscope, but its limitations in this field were quickly revealed. In view of the widespread interest in the subject, the Institute of Physics undertook to convene a Conference at which the whole matter could be explored and discussed, and the present volume comprises the papers read thereat, and the discussions which ensued.

The subject is wide, as has been indicated, and in its mathematical aspects deep, but it has its lighter moments, as in the case of the ingenious experimenter who took spiders to sea on a weather ship, so that they might furnish webs which, arranged on a wire frame and rotated on the crew's gramophone, swept the suspended salt particles from the ocean air for him to count at his leisure. Inevitably, at the opposite extreme of complexity, automatic methods for particle sizing and counting, by means of electro-optical scanning and electronic counting systems, are heavily engaged, and, for once, do not emerge from the encounter with that glamour of semi-perfect achievement which one is nowadays accustomed to expect from such devices. In fact, achievement in such a complex and difficult field must necessarily at this stage be hard-won, but a very solid foundation has been laid, both in theory and in practice, and future workers, in particular those whose interests lie in any field involving particulate suspensions and their physical behaviour, will find in the present volume a basis for their theorising and experimentation which should serve them well. To such it can be thoroughly recommended.

N. H. CHAMBERLAIN

Les Applications pratiques de la Luminescence

By M. Déribéré. 3rd edition 1955. Pp. viii + 399. Paris: Dunod. Price, 3300 francs.

The appearance of the third edition of M. Déribéré's book indicates the interest taken everywhere in the practical applications of fluorescence.

Twenty-eight chapters deal with the technique of the subject, the examination of inorganic and organic materials, its use in chemistry, physiology, biology, agriculture, industrial chemistry, and the food and drink industries; the examination of oils, lubricants, resins, paints, plastics, rubber, paper, textiles and dyes, tanning agents, perfumery, constructional materials, and museum exhibits.

Daylight-fluorescent pigments and dyes are discussed, and also the fluorescent brightening agents. A short chapter is devoted to each of these subjects, and will be of general interest to readers of this *Journal*, who will also refer to the chapters on textiles and dyeing, and to those chapters relating to organic chemistry connected with their field of work. The writer has the feeling, however, that members of our Society who have had some previous experience of this subject will find little, if anything, new. The latest reference that could be found in the new chapter on fluorescent brightening agents is dated 1953. Throughout the book references to papers later than 1946 number just over 100, and of these only about half are later than 1950, and are confined to the chapters on textiles and dyes, "optical bleaches", daylight-fluorescent colours, fluorescent colours viewed by daylight, decoration and publicity, luminescent X-ray screens, and illumination using ultraviolet radiation. This rather suggests that revision has been cursorily performed, and there are several indications of poor proofreading.

The bibliography of books at the end lists only the early editions of several well known books, whilst others are omitted altogether, and the author appears to have adopted no consistent form for giving references. The book will be of value to those who require an introduction to the subject, and in this respect adequately fulfils one of the objects of the book, namely to stimulate interest in this field, but the specialist, or one knowledgeable in this subject, will find little to reward him.

J. A. RADLEY

The Modern Textile Dictionary

By G. E. Linton. Pp. xxii + 772. New York: Duell, Sloan & Pearce–Little, Brown & Co. 1954. Price, $ 12.50.

A technical dictionary is judged by its treatment of terms with which we are familiar, and consulted for information regarding those with which we are

unfamiliar. The dyer and colourist will therefore judge it by its definitions of the terms in everyday use in his work, and acquire confidence in it only if it measures up to his standards. One wonders whether the author of *The Modern Textile Dictionary* took these facts sufficiently to heart and co-opted a staff of helpers of sufficiently wide experience collectively to satisfy all sections of the textile industry, even though he claims that more than sixty industrial firms and thirty outstandingly qualified experts have given freely of their specialised knowledge. They have needlessly digressed by including in the 12,000 terms non-technical subjects such as management and labour, the time devoted to which would have been better employed in tidying up and adequately cross-referencing essential terms. There are "scabs" in this country—they used to be blacklegs, and tomorrow they may be something else—but the word appears complete with definition in this volume, though a dictionary of slang would be a more appropriate source of information. An industrial strike and a sympathetic one are both defined, but the reader will look in vain for a definition of *strike* as applied to the initial stages of dyeing a textile fibre.

The style is sometimes perverse, a typical example being "FLUORESCENCE: When pigments glow for 1/10,000th of a second after exposure to light, they are said to be fluorescent. Pigments of this type must, therefore, be exposed continuously to stimulus".

To take other cases almost at random, the definitions of Vat Dyes, Direct Dyes, Discharge Printing are either misleading or inadequate, and the present reviewer was distressed to find that S.R.A. Dyes meant sulpho-resinic alcohol dyes.

Turning to the textile fibres, one finds that Dacron is adequately dealt with, and is identified with Terylene. The latter, however, is dealt with most inadequately, and without cross-reference to Dacron. Nylon is described correctly as a synthetic linear polymer. Under "Polymers", however, it is given as an example of a copolymer. It is amusing, but scarcely instructive, to read that the raw materials which may be used for Orlon include coal, air, limestone, natural gases, petroleum, and water; though, in fairness, it is classed as an acrylic fibre.

The chemical formula of cotton is given as "$C_6H_{10}O_5$, taken n times. The n is what makes cotton capable of taking high twist, as well as being spun into fine yarn counts . . .". Helpful?

The reader will find much to interest and amuse him, including nine pages devoted to the United States Government list of Cotton Fabric Uses, but he is advised not to neglect entirely the work of the Terms and Definitions Committees of this Society and of the Textile Institute.

H. C. Olpin

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University

Badische Anilin- und Soda-Fabrik

Helizarin Matt White and Matt Coloured Effects in Screen Printing— This card contains eight direct prints on viscose rayon, acetate rayon, silk, and Perlon illustrating the use of Helizarin White T alone and in mixtures with various Helizarin pigment printing colours. In six instances the prints have been produced on undyed grounds, but a white and a pale blue print on a black ground have been included to demonstrate the possibility of obtaining white and coloured effects on dark grounds by direct printing instead of the normal white or coloured discharge method.

Indanthren, Anthra, and Brilliant Indigo Dyes: Machine Prints on Cotton Fabrics— Direct prints of 38 Indanthren, 2 Anthra, and 6 Brilliant Indigo dyes are included in this card. The text includes methods of application, fastness figures, and details of recommended uses for each dye.

The Geigy Co. Ltd.

Cuprophenyl Brown 6GL— This direct dye gives bright yellow-browns on cellulosic fibres when after-treated on the fibre with copper sulphate, and is recommended for use alone and in mixtures where good fastness to light and washing is demanded. It is also of interest for dyeing the cellulosic component of wool-cellulosic fibre mixtures. Fastness figures for an aftercoppered dyeing on cotton include— Light 6–7, washing *b* (60°c.) 4–5, perspiration 4.

Irgalan Blue GL— This neutral-dyeing premetallised dye is similar in dyeing characteristics to, and gives dyeings of the same order of fastness as, other members of the Irgalan range. High fastness to acid chlorination is a special feature. It is, of course, of particular interest on wool, silk, and polyamide fibres, and may be used in combination with all Irgalan and Irganol S dyes. Fastness figures on wool include— Light 6, heavy milling 4, potting 3.

Irgalan Brown 7RL— This neutral-dyeing premetallised dye gives distinctly reddish browns on wool, silk, and polyamide fibres, and is particularly recommended alone and in mixtures where dyeings of high fastness to light and wet treatments is required. It is also of interest for Vigoureux and direct printing. Fastness figures on wool include— Light 6–7, heavy milling 4–5, carbonising (neutralised) 4–5.

Irgalan Violet 5RL— This further addition to the Irgalan range gives reddish violets on wool, silk, and polyamide fibres. On wool it is recommended for dyeing loose fibre, slubbing, yarn, and also cloth provided that adequate preparation is given and strict control is exercised during dyeing. It is also suitable for direct printing on these fibres. Fastness figures on wool include— Light 6–7, washing *b* 4–5, perspiration 5.

Irganol Yellow 5GLS— This neutral-dyeing acid dye gives very bright, pure yellows and is primarily intended for use in combination with members of the Irgalan and Irganol S ranges for dyeing and printing wool, silk, and polyamide fibres. Fastness figures on wool include— Light 6, heavy milling 4, carbonising (neutralised) 4.

Irganol Yellow 5GLS in combination with Irganol Green BLS— This card contains dyeings of the two dyes in five depths, alone and combined in three different proportions to give a range of greens of very good fastness to light and washing.

SOLOPHENYL TURQUOISE BLUE GRL— This direct dye gives very bright, pure blues on cellulosic fibres, slightly redder in hue than those given by the GL brand, and somewhat faster to washing. It is very level dyeing and covers "barry" viscose rayon well. Dyeings have excellent fastness to light and may be crease-resisted provided that a catalyst is used which contains zinc chloride. It is also of interest for dyeing silk and wool–cellulosic fibre mixtures and for direct printing. Fastness figures on cotton include— Light 6–7, washing 3, perspiration 2.

Imperial Chemical Industries Ltd.

HEXAGON DIGEST No. 21— This issue contains the following papers— *Slime Control in the Paper Industry* by H. E. Cook, *The Application of Colorimetry in Dyeing Research* by A. N. Derbyshire, and *Quantitative Methods for the Study of Vat Printing Processes* by W. F. Liquorice.

TECHNICAL INFORMATION LEAFLETS— *Dyehouse No. 246. Quintolan W: Application to Terylene Polyester Fibre and Nylon Fabrics*— It is recommended that, when this product is used for producing a water-repellent finish on Terylene and nylon, the impregnated fabric should be dried and then baked for 5 min. at 150°C.

Dyehouse No. 247. The Dyeing of Nylon Hose— Details are given of an investigation to determine the most suitable disperse dyes for dyeing hose to be postboarded by either steam or dry heat. With the recommended dyes no trouble is likely to arise due to dye migration, provided that saturated steam is supplied to the postboarder and no water is allowed to condense on the hose. An additional note describes a method of removing copper contamination arising from the use of needles with a high copper content. It is recommended that the hose are scoured using Lissapol NC, treated in 0·1% nitric acid for 20–30 min. at 60°C., and then rinsed in 0·05% ammonia solution.

Dyehouse No. 250. Subject Index to Technical Information Leaflets (Dyehouse) No. 161–249.

Dyehouse No. 251. The Dyeing of Strong Fibro (Courtaulds' High-tenacity Viscose Staple)— Observations are made based on dyeings produced on a 2/80s cotton-count yarn, filament denier 1·25 and staple length 1¼ in., and on fabric woven from strong Fibro yarns. The build-up, levelling power, and exhaustion of the dyes examined are similar to those obtained on normal Fibro yarn. When winch dyeing, care should be taken to avoid rope marks. With tightly woven cloths improved results should follow preparation in open width, and pad–dyeing methods are recommended.

Dyehouse No. 252. Foodstuff Colours: Permitted Lists— This leaflet contains amendments to the lists of countries in which the different dyes described in *Edicol Supra Colours for Foodstuffs* are permitted.

AFFINITY EFFECTS DURING PADDING— Reprint of a lecture by W. J. Marshall (J.S.D.C., **71**, 13 (Jan. 1955)).

THE BEHAVIOUR OF LEUCO VAT DYES ABOVE 100°C.— Reprint of a lecture by S. Blackburn and M. R. Fox (J.S.D.C., **71**, 80 (Feb. 1955)).

SOME FACTORS IN THE MEASUREMENT OF AFFINITIES OF VAT DYES FOR CELLULOSE— Reprint of a paper by R. H. Peters and J. Simons (J.S.D.C., **70**, 557 (1954)).

DYES FOR TERYLENE LOOSE STAPLE, SLUBBING, AND YARN— This card contains 20 dyeings on Terylene 4·5-in., 4-denier staple fibre carried out at 120°C. in a circulating-liquor machine. The majority have been produced with the five disperse dyes which are most suitable where good fastness to light, washing, decatising, and hot pressing is required. The card includes also a bright red produced by the modified azoic technique using a one-bath process at 100°C. and a navy and a black produced in the same way by a two-bath method.

BRENTOGEN RED VIOLET 2R— This stabilised azoic composition gives deep red–violets on cellulosic fibres. Applied by the normal caustic alkali method, it may be developed by acid ageing or in a hot, dilute organic acid bath; while it may be developed by neutral steaming if applied by the Brentogen Developer N method. Fastness figures on cotton include— Light (6% depth) 5–6, washing (repeated severe, 5 times at 100°C.) 3–4, hypochlorite ("dry chemicking") 4.

QF DURINDONE PRINTING BLACK BL; TL— The prefix QF denotes that these vat dyes have been specially prepared to ensure very rapid fixation on cotton and viscose rayon when applied by the potassium carbonate–Formosul process, since they are less sensitive to variation in concentration of alkali and reducing agent in the print paste and are less affected by adverse steaming conditions. They are supplied in a non-drying, non-settling form, and, having a low freezing point, may be stored at low temperatures. They give grey to black prints of excellent fastness to light and are recommended in all cases where freedom from accelerated degradation on exposure to light is essential. They may also be applied by the pad–steam method and are suitable for grey or black discharges. Fastness figures on cotton include— *QF Durindone Black BL:* Light 7, hypochlorite ("dry chemicking") 5, washing (Test No. 5, 5 times at 100°C.) 3–4. *QF Durindone Printing Black TL:* Light 7, hypochlorite ("dry chemicking") 5, washing (Test No. 5, 5 times at 100°C.) 3–4.

CALEDON OLIVE MW— This vat dye gives olive greens on cellulosic fibres intermediate in hue between those given by Caledon Olive Green B and Olive D. Since it is rapidly reduced, it is particularly suitable for jig dyeing, the pad–steam process, and the Standfast molten-metal process. All brands are suitable for leuco-vat dyeing; Paste Fine and FD Powder Fine brands are recommended for pigmentation processes. It is also of interest for dyeing silk. Fastness figures on cotton include— Light 7, soda boiling (open kier)–hypochlorite bleaching 3–4.

CHLORAZOL DIAZO ORANGE 3G— This direct dye (Group C) gives bright oranges on cellulosic fibres when diazotised and developed on the fibre with Direct Developer Z. In addition to the expected good fastness to washing, dyeings are faster to light than those obtained with other diazotisable yellows and oranges. It is particularly suitable for dyeing rayon staple cloths which are subsequently to be crease-resisted, since fastness to light and washing is improved thereby and the hue is practically unaltered. White discharges on developed grounds are readily obtainable using neutral Formosul or Redusol Z. It is also of interest for dyeing silk. Fastness figures for a dyeing on cotton aftertreated with Direct Developer Z include— Light 5, washing (Test No. 2, once at 55°C.) 3, perspiration 4.

DURANOL BROWN BT— This disperse dye is particularly recommended, alone and in mixtures, for dyeing all forms of acetate rayon where good fastness to light and repeated mild washing is required. It is of very little interest for dyeing nylon and of no interest on Terylene or polyacrylonitrile fibres. Fastness figures on acetate rayon include— Light 6 (bright acetate rayon), perspiration 4, washing (Test No. 2, once at 55°C.) 3.

DURAZOL BRILLIANT BLUE B— This direct dye (Class B) gives bright blues on cellulosic fibres and is recommended, alone and in mixtures, for producing full dyeings on cotton and rayon furnishings and dress material. Urea–formaldehyde finishing reduces the light fastness by one grade, but at 5–6 it is still good. A CR quality is available when reservation of acetate rayon is required. The BP brand is of interest for direct and discharge printing, its resistance to Formosul and Redusol Z making it valuable as an illuminating dye. Fastness figures on cotton include— Light 6, washing (Test No. 1, once at 40°C.) 2, water 2.

THIONOL SKY BLUE 6BMS— This solubilised sulphur dye gives bright blues on cotton and is recommended for dyeing loose fibre and yarn packages in circulating-liquor machines, for continuous warp dyeing, and for non-bronzed dyeings on cloth. Aftertreatment with acid dichromate improves the wet fastness, and peroxy compounds give increased brightness. Fastness figures on cotton include— Light 4–5, washing (repeated mild, 5 times at 40°C.) 3, perspiration 3.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

PATENTS

Carbon Black. United Carbon Co. Inc. (Maryland). *USP* 2,682,450

Apparatus which enables better control of the quality and yield of carbon black produced by decomposition of gaseous or fluid hydrocarbons. C. O. C.

Drying Yarn Packages. E. A. Stienen. *USP* 2,686,977

Apparatus in which the packages are extracted and dried in the same chamber. C. O. C.

Winch Machine. Celanese Corpn. of America. *USP* 2,684,586

In a machine having an elliptical winch and a circular idler the fabric in rope form is guided from the vat below the discharge end of the winch and across the front of the machine for re-threading on the winch, by simple means which avoids imparting tension to the fabric and/or abrading or chafing it. C. O. C.

Treating Fabrics with Liquids. Van Vlaanderen Machine Co. *USP* 2,684,887

Two elliptical winches are placed at either end of a tank. The fabric travels spirally about the winches and when it reaches the far end of one of them it passes to the other winch. C. O. C.

Centrifuge. Thomas Broadbent & Sons. *BP* 732,198

A centrifuge which, even if badly loaded so that the load is much out of balance, functions satisfactorily at high speeds without undue stress in the machine. Special foundations are thus unnecessary and there is no liability to gyroscopic precession of the cage containing the materials. C. O. C.

Weft Straightening Apparatus. Mount Hope Machinery Co. *USP* 2,682,094

In a weft straightener of the tilted roller type lateral crawling of the fabric in one direction along a tilted roll is neutralised by equal lateral crawling of the fabric in the opposite direction along another tilted roll. C. O. C.

Treating Fabric with Superheated Steam. J. Dungler. *USP* 2,682,116

In a drying machine using superheated steam as the drying medium a depression or vacuum effect is produced in the zone of the blowing nozzles, so that the velocity of the steam in that zone may be varied in accordance with the suction produced by the exhaust fan for the used steam. C. O. C.

Continuous Cylindrical Printing Screens. Tintoria Comense. *BP* 732,614

A seamless tubular screen is placed on a cylindrical support, coated with photosensitive material, dried and the screen formed by the usual photographic process, all these processes being carried out while the screen is on the support. C. O. C.

Screen Printing. J. Bley. *BP* 732,742

The stencils are carried by an endless chain conveyor which brings each in turn to the printing position. C. O. C.

Nylon Hose Finishing Machine. S. Bailey. *BP* 729,949

The hose are placed on metal forms mounted in groups on trucks passing through a compartmented autoclave containing steam at super-atmospheric pressure. Entry and exit of the trucks are accomplished through chambers in which the pressure is equalised before the autoclave is opened. During passage through the autoclave, plasticisation is followed by scouring and dyeing in which the liquors are sprayed on to the hose. G. E. K.

Apparatus for Drying Leather without the Use of Paste. United Shoe Machinery Corpn. *USP* 2,686,976

Mechanical Electrostatic Charging of Fabrics for Air Filters (VI p. 559).
The Barotor in Practical Application (VIII p. 561).

II—WATER AND EFFLUENTS

Biological Oxygen Demand (B.O.D.) of Textile Chemicals. AATCC Rhode Island Section Subcommittee on Stream Pollution. *Amer. Dyestuff Rep.*, **44**, P 355–P 359 (23 May 1955).

The 5-day B.O.D. of 64 textile chemicals and dyes is determined, and that of 90 others compiled from the literature. Results are tabulated. J. W. B.

Bleachcroft and Dyehouse Waste Studies. J. L. Brown. *Amer. Dyestuff Rep.*, **44**, P 385–P 386 (6 June 1955).

Details are given of a system whereby dye and bleach effluents are segregated into three components—the acid waste, the strongly alkaline waste, and the mildly alkaline waste. The first two are stored and blended through a settling tank and filter or filters, the effluent being combined with mildly alkaline waste and domestic sewage in proportions suitable for biological treatment. J. W. B.

Cotton Slashing with Synthetic Compounds as a means towards Pollution Abatement. D. W. Snyder. *Amer. Dyestuff Rep.*, **44**, P 382—P 384 (6 June 1955).

Biological oxygen demand (B.O.D.) values of synthetic products such as methylcellulose, carboxymethylcellulose, and polyvinyl alcohol are so very much lower than those of natural sizing agents that their use in cotton slashing greatly reduces stream pollution. Details of plant-scale experiments are given. J. W. B.

Biological Treatment of Mixtures of Highly Alkaline Textile Waste and Sewage. R. H. Souther and T. A. Alspaugh. *Amer. Dyestuff Rep.*, **44**, P 390–P 395 (6 June 1955).

Industrial wastes with high alkalinity are successfully treated by a trickling filter followed by activated sludge. About 93% B.O.D. (biological oxygen demand) removal is obtained, and the highly alkaline nature of the waste prevents sulphide odours. J. W. B.

Textile Waste Treatment in Texas. D. F. Smallhorst. *Amer. Dyestuff Rep.*, **44**, P 386–P 389 (6 June 1955).

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Surface-active Agents used in Textile Applications. C. Pacifico and S. Giers. *J. Amer. Oil Chem. Soc.*, **32**, 231–235 (April 1955).

Statistics and data are given of the use of surface-active agents in textile processing in the U.S.A. P. G. M.

Phase Studies of the Systems Soap–Sodium Chloride–Water. P. C. Blokker. *J. Amer. Oil Chem. Soc.*, **32**, 216–220 (April 1955).

The phase behaviour of ternary mixtures of various synthetic soaps, sodium chloride, and water is described. Several mixed sodium alkyl sulphates were investigated in detail at two temperatures; pure alkyl sulphates and alkyl and alkaryl sulphonates were investigated only tentatively. The phase relations showed a striking resemblance to those of fatty acid soaps. P. G. M.

PATENTS

Improving the Stability of Lather of Synthetic Detergents. Unilever. *BP* 732,898

Addition to organic ionic soapless detergents, e.g. the glycol and polyglycol ethers of acylphenols described in *BP* 470,181, of $> 20\%$ of a compound of formula $R^1{\cdot}CO{\cdot}Ar{\cdot}OZ$ ($R^1{\cdot}CO$ = aliphatic acyl group of 6–10 C; Ar = benzene or naphthalene; Z = H or $-R^2OH$; R^2 = alkylene, dialkylene ether, or polyalkylene polyether of < 16 C (not more than 5 C being in any one uninterrupted chain) and < 5 O), e.g. *p*-caprylresorcinol mono-α-glycerol ether, increases the stability of the lather of the detergents, although such compounds have no detergent or lathering power themselves. C. O. C.

Detergent. Unilever. *BP* 733,416

Addition of a compound of formula R^1·Ar·O·X (R^1 = Alk or acyl of 4–10 C; Ar = benzene; X = H or R^2OH; (R^2 = alkylene or subst. alkylene, dialkylene ether or polyalkylene polyether of $\geq$ 15 c ($\geq$ 4 of which are in any uninterrupted chain) and < 5 O), e.g. caprylyl resorcinol α-monoglycerol ether, to a synthetic anionic detergent being added to soap to prevent coagulation of lime soaps considerably reduces the amount of synthetic detergent needed. C. O. C.

Improving the Lathering Properties of Organic Ionic Soapless Detergents. Unilever. *BP* 733,075

Compounds of formula R^1·(Ar)$_n$·O·R^2·CO·NH_2·CO·NH_2 (Ar = benzene; n = 0 or 1; R^1 = Alk of 4–12 C when n = 1, at least 4 being in a straight chain connected to the benzene ring, or of 8–16 C when n = 0, at least 8 being in a straight chain connected to the ether link; R^2 = alkylene or alkylidene of < 7 C), e.g. *p-tert.*-octylphenoxyacetamide, added to organic ionic soapless detergents impart increased lathering properties to the detergents. C. O. C.

Textile Lubricant. Boehme Fettchemie. *BP* 732,372

An improved fibre lubricant which is water-soluble and free from fatty and/or mineral oil components comprises (*a*) a component which causes the fibres to adhere to one another, e.g. a sugar or an ether or ester thereof, a dextrin, sulphite liquor or a polypeptide; (*b*) a viscous or viscosity-increasing component which lubricates the fibres in quantity insufficient to loosen the fibres, e.g. a polyglycol, a polyoxide wax or its water-soluble derivative, or a surface-active agent such as ammonium oleyl sulphate; and (*c*) a component which enhances the moisture-retaining capacity of the fibres, e.g. a polyhydric alcohol such as glycerol or a glycol or its water-soluble ether.

J. W. B.

Textile Lubricants. Celanese Corpn. of America. *BP* 732,933

A lubricant particularly suitable for cellulose acetate yarns, and also for yarns of other organic derivatives of cellulose, comprises an alkyl phenol, preferably diamyl phenol (2–5% by wt.), a lower aliphatic acid ester of a vegetable oil which is a glyceride containing the radical of a hydroxylated higher fatty acid or a lower aliphatic ester of a lower alkyl ester of a hydroxylated fatty acid, e.g. butyl acetyl ricinoleate (2–6%), an oxidised vegetable oil such as peanut oil (5–25%), a higher fatty acid e.g. oleic acid (2–10%), an amine, preferably an alkanolamine (3–20%), and an alkyl phosphate, e.g. phosphate ester of mixed decanols (4–20%). The remainder, e.g. about half, is mineral oil with which the others are blended.

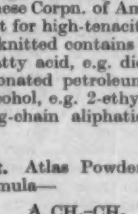
J. W. B.

Lubricant for High-tenacity Regenerated Cellulose Yarns. Celanese Corpn. of America. *USP* 2,684,311

A good lubricant for high-tenacity regenerated cellulose yarns to be warp knitted contains an alkylolamine amide of a long chain fatty acid, e.g. diethanolamine amide of lauric acid, sulphonated petroleum, a polyhydroxy long chain aliphatic alcohol, e.g. 2-ethyl-1:3-hexanediol, and a monohydroxy long-chain aliphatic alcohol, e.g. 2-ethyl-hexanol. C. O. C.

Antistatic Agent. Atlas Powder Co. *USP* 2,683,101

Compounds of formula—



(R = long chain aliphatic radical of 11–20 C; A = anion), e.g. cetyl ethyl morpholinium ethosulphate, are excellent, antistatic agents for textiles. They are applied from aqueous solution so as to leave 0·4% of the compound on the fibre. C. O. C.

Textile Sizes for Synthetic Linear Polymers. British Nylon Spinners. *BP* 732,911

Sizes which can be applied in one phase, before cold-drawing if desired, and which will not flake off during subsequent processing, e.g. knitting, comprise aqueous solutions of one or more solid high mol. wt. straight chain organic compounds of m.p. > 40°c., which are poly-alkylene glycols or condensates thereof with higher aliphatic alcohols or acids, e.g. polyethylene glycol monostearate, together with one or more hygroscopic liquid polyhydric alcohols or hydroxyamines of b.p. > 150°c. which are themselves capable of dissolving little or none of the above solid organic compounds. When the size is dried the deposit consists of a mixture of solid and liquid phases, the former comprising 20–80% by wt.

J. W. B.

Increasing the Viscosity of Carboxymethylcellulose Solutions. S. C. Johnson & Son. *USP* 2,684,914

The viscosity of aqueous solutions of carboxymethyl-cellulose is increased by adding < 1% (by wt. on the solution) of formaldehyde and < 1% of NaOH or Na *o*-phenyl-phenoxide. C. O. C.

Size for Glass Fibres. Owens-Corning Fiberglas Corpn. *USP* 2,684,309

A mixture of 30–50% by wt. of terphenyl and 70–50% of hydrogenated terphenyl has excellent adhesion both to glass fibres and to the various overcoats or finishes applied to the fibres. C. O. C.

Non-foaming Surface-active Alkyl Benzene Sulphonate Composition. Monsanto. *USP* 2,683,692

Combinations of an alkylbenzene sodium sulphonate (Alk of 8–14 C) with compounds of formula $CH_3COOH·NH_2R$ (R = Alk of 14–16 C) are nonfoaming and surface active. Thus addition of 0·2% of a mixture of 91·5% of decylbenzene sodium sulphonate and 8·5% of the acetic acid salt of myristylamine to a boiling dyebath acts as an excellent levelling agent. C. O. C.

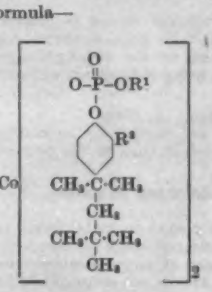
Water-dispersible Emulsions of Polysiloxanes for imparting Water-repellency to Nylon and Similar Synthetic Hydrophobic Fibres; Textile Lubricants and Softening Agents. DuP. *USP* 2,702,276

The emulsions made by mixing a polysiloxane of formula—

$(CH_3)_3SiO(-Si(CH_3)H·O)_n Si(CH_3)_3$ or $R_2SiO(-SiR_2·O)_nSiR_2$ (n = 1–500; R = Alk of < 4 C) with glycerine and a dispersing agent readily disperse in water to give stable dispersions. Dispersions containing compounds of the first type are suitable for giving a hydrophobic finish to nylon etc. by impregnation and then heat curing. Dispersions containing the second type of compound are useful as textile lubricants or softening agents. C. O. C.

Ester-salts of Phosphoric Acid— Driers and Wetting Agents in Non-aqueous Liquids, Plasticisers, etc. ICI. *BP* 732,561

Compounds of formula—



(R^1 = Alk of < 13 C; R^2 = H or Alk), e.g. cobaltous *p*-tertiary octylphenylethyl phosphate, may be used as wetting agents for pigments in non-aqueous liquids, driers for drying oils, surface tension reducing agents and plasticisers. C. O. C.

Solvent for Polyacrylonitrile. Rottweiler Kunstseidefabrik. *BP* 732,117

Solutions of polyacrylonitrile in perchloric acid are useful for spinning into fibres etc.; the rate of hydrolysis is much lower than in sulphuric acid. W. G. C.

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

p-Xylene from Petroleum. H. W. Haines, Jr., M. Powers, and R. B. Bennett. *Ind. Eng. Chem.*, **47**, 1096–1103 (June 1955).

Colour Reactions of Unsaturated Carbonyl Compounds. II—Use of p-Aminodimethylaniline. S. Hünig, J. Utermann, and G. Erlemann. *Chem. Ber.*, **88**, 708–712 (May 1955).

Steric Hindrance in Alkyltrinitrophenols. C. E. Moore and R. Peck. *J. Org. Chem.*, **20**, 673–679 (May 1955).

The dissociation constant of picric acid is reduced to about one-hundredth of its value by a methyl or ethyl substituent, and to even less by two methyl groups, or a methyl and an ethyl, or a single *iso*propyl group. Effects on the ultraviolet absorption spectrum are also studied.

H. E. N.

Physicochemical Properties of Hydrophobic Dyes. I—Conformity of Dyes with Bouguer's Dilution Law [Beer's Law]. E. A. Veller and B. A. Porai-Koshits. *J. Appl. Chem. U.S.S.R.*, **28**, 497–506 (May 1955).

Absorption spectra are determined over the range 250–750 mμ. for soln. of the following oil-soluble and disperse dyes in dichloroethane at concn. ranging from $\sim 10^{-4}$ to $\sim 10^{-2}$ g./ml.—Yellow G fat-soluble (2:4-xylidine→3-methyl-1-phenylpyrazol-5-one), Scarlet G for acetate rayon (*p*-nitroaniline→*N*-ethyl-*N*-2-hydroxyethylaniline), Sudan Red 7B (aminoazobenzene→*N*-ethyl-2-naphthylamine), Fat-soluble Blue (1-methylamino-4-*p*-tolylaminoanthraquinone), Blue K for acetate rayon (1-(2-hydroxyethyl)amino-4-methylaminoanthraquinone), and 1:4-dihydroxy-5:8-bis-*p*-tolylaminoanthraquinone. The departures from Beer's law at the highest concn. examined do not exceed 5–6% at the visible absorption max. and, in the preparation of light filters from these dyes, it may normally be assumed that the law is obeyed exactly. For dyes containing OH groups there are considerable departures from the law in the ultraviolet region, an effect that is considered to be associated with increased intermolecular hydrogen bonding at higher concn.

A. E. S.

Kinetics of the Reduction of Azoxybenzene (and Some Derivatives) by Titanous Chloride. M. J. Stephen and C. Hinshelwood. *J.C.S.*, 1393–1406 (May 1955).

Reduction of azoxybenzene (I) by $TiCl_3$ in aq. alcohol proceeds as follows: (*a*) I → aniline; (*b*) I → azobenzene → hydrazobenzene → benzidine + aniline; ca. 20% of the total follows (*a*). In (*b*) the velocity constants of the first and second stages are of comparable magnitude, and the azobenzene concn. passes through a max. The initial rate of reduction of I is proportional to $1/[H^+]$, $[TiCl_3]$, and $[C_6H_5{\cdot}NO{:}N{\cdot}C_6H_5]$, and the whole system exemplifies second-order consecutive reactions. The influence of substituent groups on the rate appears to be considerably smaller than in the reduction of substituted nitrobenzenes. The large differences in the effects of acid concn. on the $TiCl_3$ reduction of nitrobenzene, I, and azobenzene are discussed. The fact that the reduction of I by $TiCl_3$ gives some benzidine, whereas that of nitrobenzene gives 100% aniline, re-opens a question about the course of the latter reduction.

H. H. H.

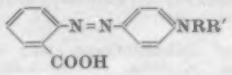
"Diazoperbromides". M. Aroney and R. J. W. LeFèvre. *J.C.S.*, 1630 (May 1955).

The infrared absorption spectra between 3250 and 1350 cm.$^{-1}$ of the diazoperbromides from *p*-toluidine, *p*-chloroaniline, and β-naphthylamine indicate the presence of the $R{\cdot}N_2^+$ group (cf. Hantzsch, *Ber.*, **48**, 1344 (1915), and Forster, *J.C.S.*, **107**, 260 (1915)). The relative stability of solid diazonium perbromides is suggested as being due to the shapes of their cations and anions, which permit a close mutual approach so that electrostatic forces are reinforced by van der Waals forces, thus favouring the stability of an ion-pair arrangement.

H. H. H.

Spectra of o-Carboxyazobenzene Derivatives. W. C. J. Ross and G. P. Warwick. *Chem. and Ind.*, 745–747 (25 June 1955).

The absorption maximum of I in 95% aqueous ethanol changes with change of substituents in the order: R = R′ = $CH_2{\cdot}CH_2Cl$, λ_{max} = 470 mμ. and 410–430 mμ.; R = C_2H_5, R′ = $CH_2{\cdot}CH_2Cl$, λ_{max} = 485 and 415–445 mμ.; R = R′ = CH_3, λ_{max} = 498 mμ. This is associated with increasing proportions of a resonance hybrid II and, together with the absence of the O–H stretching band (3600 cm.$^{-1}$), is regarded as evidence for the existence of a hydrogen bond between carboxyl and α-nitrogen in I.



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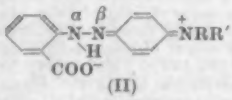
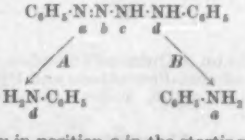


(II)

A. J.

Reactions with ^{15}N. XVII—Formation of Phenyl Azide from Benzenediazonium Chloride and Phenylhydrazine Hydrochloride. K. Clusius and H. Craubner. *Helv. Chim. Acta*, **38**, 1060–1065 (June 1955).

The reaction probably proceeds thus—



Using ^{15}N only in position *a* in the starting products, it is shown that none of it appears in positions *b* or *c* of the intermediate, and that the reaction proceeds by routes *A* and *B* in the ratio 46 : 54. In hydrochloric acid solution no exchange occurs between $[C_6H_5{\cdot}{}^{15}NN]^+Cl^-$ and aniline.

H. E. N.

Formazyl Complexes. M. Seyhan. *Chem. Ber.*, **88**, 646–648 (May 1955).

The formazan obtained by coupling diazotised anthranilic acid with the phenylhydrazone of benzothiazole-2-aldehyde forms a dark red crystalline 2 : 1 complex with uranyl acetate. The formazan obtained from diazotised aniline would not form a complex.

H. E. N.

Iminazopyridines and Related Ring Systems. I—Syntheses of Iminazopyridines. I. K. Schilling, F. Kröhnke, and B. Kickhöfen. *Chem. Ber.*, **88**, 1093–1102 (July 1955). **II**—F. Kröhnke and B. Kickhöfen. *Ibid.*, 1103–1108. **III—The Iminazothiazole Ring System.** B. Kickhöfen and F. Kröhnke. *Ibid.*, 1109–1114. **IV—Iminazoquinoline.** B. Kickhöfen. *Ibid.*, 1114–1116. **V—Orientation of Iminazopyridines.** F. Kröhnke, B. Kickhöfen, and C. Thoma. *Ibid.*, 1117–1121.

Bromination of Pyrazoles. R. Hüttel, H. Wagner, and P. Jochum. *Annalen*, **593**, 179–200 (June 1955). **Iodination of Pyrazoles.** R. Hüttel, O. Schäfer, and P. Jochum. *Annalen*, **593**, 200–207 (June 1955). **Halogenation of 1:2:3-Triazoles.** R. Hüttel and G. Welzel. *Annalen*, **593**, 207–218 (June 1955).

Synthesis and Fluorescence of Quinoline-substituted 1:3:5-Triphenylpyrazolines. O. Neunhoeffer and H. Ulrich. *Chem. Ber.*, **88**, 1123–1133 (July 1955).

Pyrazolones and Pyrazole Ethers. W. Krohs. *Chem. Ber.*, **88**, 866–874 (June 1955).

The sodium salts of 1:5-dimethylpyrazol-3-one and of its 4-dimethylamino deriv. react with heterocyclic halogen cpd. to yield pyrazole ethers, but the sodium salt of the

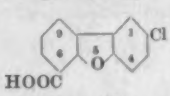
4-dimethylamino deriv. reacts with aliphatic halogen cpd. to give a mixture of the corresponding ethers and *N*-alkylpyrazolones. H. E. N.

3-Nitrobenzofuran. F. Kaluza and G. Perold. *Chem. Ber.*, **88**, 597–601 (May 1955).

This is obtained in 60% yield, together with 2-nitrobenzofuran and salicylic acid, by the action of NO_2 on benzofuran in benzene soln. at 6–10°C. The constitution is proved by reduction with $LiAlH_4$ to 3-amino-2:3-dihydrobenzofuran. Boiling water causes 3-nitrobenzofuran to split off formic acid quantitatively, forming the unknown 2-hydroxy-*ω*-nitrotoluene, identified as its known 3:5-dibromo deriv. H. E. N.

Chlorination of Dibenzofuran and some of its Derivatives. K. Oita, R. G. Johnson, and H. Gilman. *J. Org. Chem.*, **20**, 657–667 (May 1955).

Dibenzofuran is most conveniently chlorinated by means of chlorine in presence of iron powder, the 2-chloro and 2:8-dichloro cpd. being formed. The 3-chloro deriv. is obtained by the action of thionyl chloride on 3-nitrodibenzofuran at 185–190°C. Metalated (Li) 2-chlorodibenzofuran on oxidation yields the 4-hydroxy cpd., on carbonation the 4-carboxylic acid. Chlorination of 4-hydroxydibenzofuran (Cl_2–Fe) leads to the 3-chloro deriv., which is converted by sulphuryl chloride to 1:3-dichloro-4-hydroxydibenzofuran. Dibenzofuran-4-carboxylic acid on chlorination (Cl_2–Fe) yields 2-chlorodibenzofuran-6-carboxylic acid (I).



(I)

H. E. N.

Action of Light on *o*-Quinone Diazides. VI—Photosynthesis of *cyclo*Pentadiene and Pyrrole Derivatives. O. Süs and K. Möller. *Annalen*, **593**, 91–126 (June 1955).

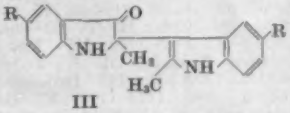
Reactions with Nitrosodisulphonates. VIII—*o*-Benzoquinones and Phenazines. H.-J. Teuber and G. Staiger. *Chem. Ber.*, **88**, 802–827 (June 1955).

The preparation is described of a series of new quinones from monohydroxybenzenes carrying 4-alkyl or 4-alkoxy substituents, yields being 70–90%. The structure is proved by absorption spectra and phenazine formation with *o*-phenylenediamine. The alkyl-substituted *o*-benzoquinones tend to polymerise in the solid state and in soln.

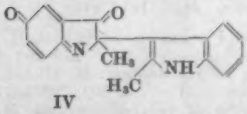
IX—Infrared Spectra of *o*-Benzoquinones. W. Otting and G. Staiger. *Ibid.*, 828–833. H. E. N.

X—Oxidation of some Indoles. H.-J. Teuber and G. Staiger. *Chem. Ber.*, **88**, 1066–1071 (July 1955).

At pH 3·5–4, 2-methylindole (I) and 2:5-dimethylindole (II) yield the yellow cpd. III (R = H and CH_3 respectively).

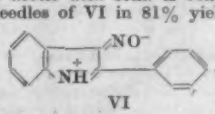


III



IV

In neutral soln., I forms a blue cpd. to which formula IV is assigned. II yields no such product, but 2-phenylindole (V) does. V in acetic acid soln. is converted into the golden-yellow needles of VI in 81% yield.



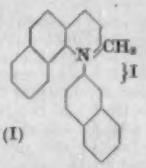
VI

H. E. N.

Colour of Iodides, Thiocyanates, and Other Salts of Certain Pyridinium Bases. F. Kröhnke. *Chem. Ber.*, **88**, 851–863 (June 1955).

Cyanine Dyes. V—Synthesis and Reactions of *N*-Arylquinaldinium Salts. G. T. Pilyugin. *J. Gen. Chem. U.S.S.R.*, **25**, 793–802 (April 1955).

The reaction described previously (see J.S.D.C., **70**, 307 (1954); **71**, 416 (July 1955)) is applied in the synthesis of other new *N*-arylquinaldinium salts, viz. 2-methyl-1-(2-naphthyl)benzo[*h*]quinolinium iodide (I), 6-hydroxy-1-phenylquinaldinium iodide, and 8-methyl-1-*o*-tolylquinaldinium perchlorate, and these are used in the synthesis of the following dyes (wavelengths are those of absorption max. in alcoholic soln.)— bis[1-(2-naphthyl)-2-benzo[*h*]quinoline]trimethincyanine iodide (648 m*μ*.), bis(6-hydroxy-1-phenyl-2-quinoline) trimethincyanine iodide (626 m*μ*.), bis(8-methyl-1-*o*-tolyl-2-quinoline)-trimethincyanine perchlorate (623 m*μ*.), bis(6-hydroxy-1-phenyl-2-quinoline)-*β*-methyltrimethincyanine iodide (620 m*μ*.), bis(8-methyl-1-*o*-tolyl-2-quinoline)-*β*-methyltrimethincyanine perchlorate (617 m*μ*.), [1-(2-naphthyl)-2-benzo[*h*]quinoline](3-ethyl-2-benzothiazole) trimethincyanine iodide (596 m*μ*.), (8-methyl-1-*o*-tolyl-2-quinoline)-(3-ethyl-2-benzothiazole)trimethincyanine perchlorate (584 m*μ*.), [1-(2-naphthyl)-2-benzo[*h*]quinoline](1:3:3-trimethyl-2-*pseudo*indole)trimethincyanine iodide (584·5 m*μ*.), (8-methyl-1-*o*-tolyl-2-quinoline)(1:3:3-trimethyl-2-*pseudo*indole)trimethincyanine perchlorate (565 m*μ*.), 2-*p*-dimethylaminostyryl-1-(2-naphthyl) benzo[*h*]-quinolinium iodide (550 m*μ*.), 2-*p*-dimethylaminostyryl-6-hydroxy-1-phenylquinolinium iodide (542 m*μ*.), and 2-*p*-dimethylaminostyryl-8-methyl-1-*o*-tolyl-quinolinium perchlorate (546 m*μ*.). Like the analogous dyes described previously (*loc. cit.*), these dyes also are deeper in colour than the corresponding dyes having ethyl in place of aryl.



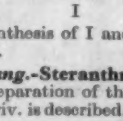
(I)

A. E. S.

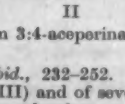
Polycyclic Quinones. II—1:4-Bisarylaminoanthraquinones. N. S. Dokunikhin and T. N. Kurdyumova. *J. Gen. Chem. U.S.S.R.*, **25**, 617–622 (March 1955).

Visible absorption spectra are determined in CCl_4 soln. for five 1:4-bisarylaminoanthraquinones (aryl = *p*-tolyl, 2:4-xylyl, mesityl, 2-bromo-4:6-dimethylphenyl, and 2:4:6-triethylphenyl) and two 1:4-bisarylamino-2:3-dimethylanthraquinones (aryl = *p*-tolyl and mesityl), and variations observed are considered in their relation to the structural, particularly steric, peculiarities of the cpd. A. E. S.

"Steranthrene". VI—*lin.*- and *ang.*-1:2-Benzoaceperinaphthan. H. Dannenberg and D. Dannenberg-von Dresler. *Annalen*, **593**, 219–231 (June 1955).



I

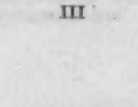


II

The synthesis of I and II from 3:4-aceperinaphthan is described.

VII—*ang.*-Steranthrene. *Ibid.*, 232–252.

The preparation of this cpd. (III) and of several of its hydro deriv. is described. It forms colourless needles, m.p.



III

93°c., solutions of which fluoresce blue. The orange–red trinitrobenzene cpd. melts at 188–189°c. The ultraviolet absorption spectrum of the hydrocarbon confirms the structure assigned to it. H. E. N.

The Melanins. B. Schmidli. *Helv. Chim. Acta*, **38**, 1078–1084 (June 1955).

Natural melanin, obtained from human hair, and various synthetic melanins are examined by chromatography, spectrophotometry, and electron microscopy. The former is shown to consist of a melanoprotein and the melanin proper. Hydrolysis converts the melanoprotein into the melanin, the mol. wt. of which is found to be about 70,000 by a diffusion study, and a mixture of amino acids, in which cystine, leucine, phenylalanine, valine, alanine, glycine, and probably tyrosine and threonine are present together with some unidentified substances. H. E. N.

Relation of Leuco Anthocyanins to Anthocyanin Synthesis. R. E. Alston and C. W. Hagen. *Nature*, **175**, 990 (4 June 1955).

It is suggested that leuco anthocyanins serve in plants as precursors to the ultimate anthocyanin. W. R. M.

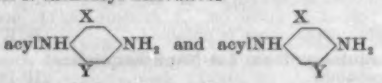
Colouring Matters of Actinomycetes. IV—Constitution of Actinorhodin and Isolation of Protoactinorhodin. H. Brockmann and V. Loeschcke. *Chem. Ber.*, **88**, 778–788 (June 1955).

The empirical formula for actinorhodin, the colouring matter of *Streptomyces coelicolor*, is shown to be $C_{14}H_{14}O_7$, comprising the naphthazarin ring system, a carboxy group, and a *C*-methyl group. In the mycelium actinorhodin is present mainly as protoactinorhodin, a reduction product. H. E. N.

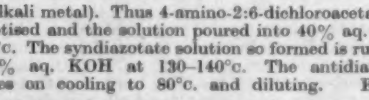
PATENTS

Antidiazotates of Substituted *p*-Phenylenediamines. FH *BP* 731,065

Antidiazotates of substituted derivatives of *p*-phenylenediamines are prepared by treating the diazo compounds of their *N*-monoacyl derivatives—



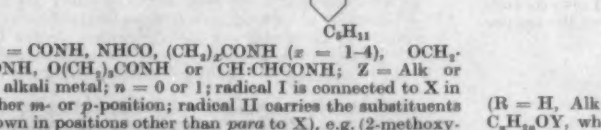
(X and Y = Hal, Alk, or OAlk; acyl = R·CO or R·SO_2 e.g. the radicals of acetic, benzoic, sulphobenzoic, or ethane sulphonic acid etc.) with cold aq. caustic alkali to give the syndiazotates, followed by heating with conc. aq. caustic alkali which hydrolyses the acyl group and gives the antidiazotates—



(M = alkali metal). Thus 4-amino-2:6-dichloroacetanilide is diazotised and the solution poured into 40% aq. KOH at > 5°c. The syndiazotate solution so formed is run into hot 80% aq. KOH at 130–140°c. The antidiazotate separates on cooling to 80°c. and diluting. E. S.

Colour Couplers. Kodak. *BP* 733,119

Compounds of formula—



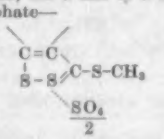
(X = CONH, NHCO, $(CH_2)_xCONH$ (x = 1–4), OCH_2·CONH, $O(CH_2)_3CONH$ or CH:CHCONH; Z = Alk or an alkali metal; n = 0 or 1; radical I is connected to X in either *m*- or *p*-position; radical II carries the substituents shown in positions other than *para* to X), e.g. (2-methoxybenzoyl)-4-[3-(3:5-dicarbomethoxyphenyl-carbamyl-valeramido)-6-(2:4-di-*tert.*-amylphenoxy) benzamido]-acetanilide, are non-migrating colour couplers which can be incorporated directly in gelatin or other hydrophilic medium. C. O. C.

Purification of 2:2′-Dimethyl-1:1′-dianthraquinonyl. DuP. *USP* 2,666,768

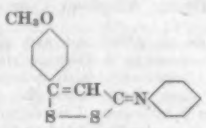
Crude 2:2′-dimethyl-1:1′-dianthraquinonyl is freed from other organic compounds and cuprous chloride by dissolving in 4–6 times its wt. of a 50/50 mixture of 95–99% H_2SO_4 and nitrobenzene, adding water until the acid strength is 81–83% and the temperature 105–110°c., filtering and washing with water. R. K. F.

1:2-Dithiol-5-thione Derivatives. A. Luettringhaus and U. Schmidt. *BP* 730,058

A trithionium salt, i.e. a salt of a 1:2-dithiol-5-thione, e.g. the methosulphate—



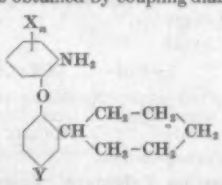
is treated with a compound containing a NH_2 group to form the corresponding 5-imido derivative. Thus 3-*p*-methoxyphenyl-1:2-dithiol-5-thione methosulphate is boiled for 2 min. in acetic acid containing aniline. The product—



is separated by adding water. R. K. F.

Red Monoazo Acid Dyes. FBy. *BP* 732,949

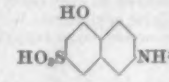
Monoazo dyes obtained by coupling diazotised amines—



(X = H, Cl, or CH_3; Y = H or *cyclo*hexyl; n = 1 or 2) with 1-acylamino-8-naphthol-mono- or di-sulphonic acids dye wool, etc. from a neutral or slightly acid bath reds of brighter hue than the similar dyes having a *cyclo*hexyl group only in the *para* position to the -O- link. These amines are prepared by reacting phenol with chloro-*cyclo*hexane to give 2-*cyclo*hexyl- or 2:4-di*cyclo*hexyl-phenol, which is fused with *o*-chloronitrobenzene or its substitution products, followed by reduction of the nitro group. Diazotisation of the aminodiaryl ethers containing two cyclohexyl radicals is carried out in solvents such as formic acid, formamide, or alcohol. Thus, 2-amino-2′:4′-dicyclohexyldiphenyl ether is dissolved in formic acid and diazotised at 0–5°c. by adding H_2SO_4 and sodium nitrite. Formamide is then added, and the diazo compound is then added to a solution of *N*-benzoyl-H acid in aqueous ammoniacal methanol. The product dyes wool bluish red from a neutral bath. E. S.

Orange Monoazo Acid Dyes. ICI. *BP* 732,121

Bright orange, level-dyeing acid dyes are made by coupling diazotised 2-amino-4-trifluoromethylbenzenesulphonic acid with J acid or its *N*-acyl derivatives—

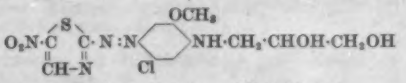


(R = H, Alk of < 6 C, alkoxy of < 5 C, or a group $C_nH_{2n}OY$, where n = 1, 2 or 3 and Y = Alk of < 5 C). The diazo component is prepared by treating 2:2′-dinitro-4:4′-di(trifluoromethyl)diphenyl disulphide with chlorine in aqueous medium, hydrolysing the 2-nitro-4-(trifluoromethyl) benzene sulphonyl chloride thus obtained, and reducing the nitro group. Diazotised and coupled with

N-acetyl-J acid in presence of sodium acetate, it yields a bright yellowish-orange acid dye. E. S.

Blue Monoazo Disperse Dyes. Eastman Kodak Co. *USP* 2,683,708

Blue monoazo disperse dyes for cellulose acetate, nylon, etc. are made by coupling diazotised 2-amino-5-nitrothiazole (which may carry a substituent in position 4) with suitable *N*-monosubstituted anilines. Thus 2-amino-5-nitrothiazole is diazotised in nitrosylsulphuric acid and coupled with *N*-$\beta\gamma$-dihydroxypropyl-5-chloro-2-methoxyaniline to give—



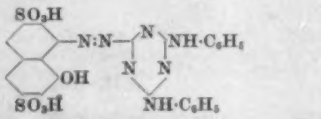
which dyes cellulose acetate reddish blue.

USP 2,683,709

Similar dyes are produced by coupling the same diazo compounds with suitable *NN*-disubstituted anilines. Thus 2-amino-5-nitrothiazole → *N*-β-hydroxyethyl-*N*-phenyl-*m*-toluidine is a dischargeable reddish-blue for cellulose acetate, etc. E. S.

Azo Triazine Dyes. American Cyanamid Co. *USP* 2,679,499

Azo compounds in which an azo group is directly attached to a C atom in a triazine ring are made by condensing a halogenotriazine with an aromatic hydrazine, followed by oxidation of the hydrazine group to give an azo group. Thus 8-hydrazino-1-naphthol-3:6-disulphonic acid (1·78 parts) is gradually added at 0°c. to an aqueous suspension of cyanuric chloride (1·01 parts), together with soda ash to destroy the liberated HCl. The solution is boiled with excess aniline, and the product oxidised with cold nitrous acid to give—



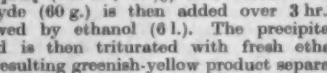
which dyes wool orange-brown. E. S.

Diazo Condensation Polymers. Ringwood Chemical Corpn. *USP* 2,679,498

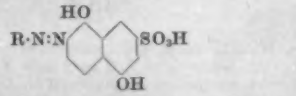
Suitable diazo compounds are reacted with formaldehyde, etc., to give polymers which find application in diazotype reproduction processes. The products are isolated as free-flowing, water-soluble powders by diluting the reaction mixtures with a water-miscible alcohol, followed if necessary by trituration with fresh alcohol. Thus *p*-diazonium diphenylamine sulphate (450 g.) is stirred into H_2SO_4 (750 cc., 98%) and cooled to 6°c. Paraformaldehyde (60 g.) is then added over 3 hr. at $\geq$ 10°c., followed by ethanol (6 l.). The precipitated toffee-like solid is then triturated with fresh ethanol (6 l.), and the resulting greenish-yellow product separated and dried to give a free-flowing powder (445 g.). E. S.

Metal(Copper)-complex Tetrakisazo Direct Cotton Dyes. S. *BP* 732,419

Similar dyes to those of *BP* 703,133 (J.S.D.C., **70**, 163 (1954)) are obtained by coupling 1 mol. of the diazo compound of an aminodisazo compound—



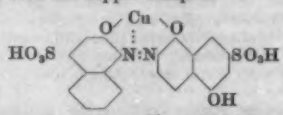
(A and B are benzene or naphthalene series radicals; the azo links are *para*- to each other; nucleus I may contain also CH_3, OCH_3, or acylamino) with 1 mol. of the copper complex of a monoazo compound (II)—



(R = the radical of a naphthol-mono-, -di-, or -trisulphonic acid, or of a pyrazolone or pyrazolonesulphonic acid, or of *p*-cresol, in which an OH group is *ortho*- to the azo link). The dyes so produced must contain > 1 SO_3H group, and may be treated in substance or on the fibre with copper compounds.

The copper complexes of the compounds (II) are obtained by coupling diazotised 2-amino-1-chloro-5-naphthol-7-sulphonic acid with RH, and heating the *o*-chloro-*o'*-hydroxyazo compound so formed with NaOH, glycerol, and a copper compound at *ca.* 90°c. for several hours.

Thus the aminodisazo compound 2-naphthylamine-6:8-disulphonic acid → *m*-toluidine → *p*-cresidine is diazotised and the suspension of diazo compound so formed coupled with the copper-complex—



in presence of pyridine. The tetrakisazo dye so formed is heated with ammoniacal copper sulphate which gives the copper complex with splitting of the methoxy group. It dyes cellulosic fibres olive. E. S.

Perinones—Dyes for Spun-dyed Melt-spun Fibres. Fran. *BP* 730,692

Perinones—

(C atoms 2 and 10 are linked to 2 or 3 other C's forming thereby a ring) made by condensing a 1:8-naphthylenediamine with an *o*- or *peri*-dicarboxylic or tetracarboxylic acid or an aliphatic 1:2- or 1:3-dicarboxylic acid, are yellow to blue dyes which will withstand the temperatures employed in melt spinning, e.g. of superpolyamides and polyesters. Thus 1:8-naphthylene diamine is heated with 3-chlorophthalic anhydride at 220–250°c. until no more water vapour is given off. A mixture of 11- and 14-chlorophthaloperinones is obtained. R. K. F.

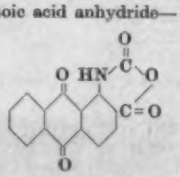
Anthraquinone from 1:4-Naphthaquinone. American Cyanamid. *BP* 731,368

The 1:4-naphthaquinone content of crude products obtained by vapour-phase oxidation of naphthalene as used to obtain phthalic anhydride, which may be as low as 1 mol. per 10 mol. phthalic anhydride, is converted to an anthraquinone by Diels–Alder reaction with an open chain conjugated diene, followed by oxidation. Thus, the product obtained by oxidation of naphthalene with air at 340°c. in contact with a fluid bed catalyst consisting of alumina and vanadium pentoxide supported on potassium pyrosulphate and silica; and containing 17·6% 1:4-naphthaquinone, 56·5% phthalic anhydride and 25·9% naphthalene, is heated at 100°c. in a steel autoclave with 1:3-butadiene and a trace of copper chloride to inhibit polymerisation. Excess butadiene is vented and recovered and the reaction mixture aerated in aq. Na_2CO_3-NaOH while simultaneously steam-distilling out naphthalene. The resulting anthraquinone is filtered off, phthalic acid being recovered by acidifying the alkaline filtrate.

R. K. F.

1-Aminoanthraquinone-2-carboxylic Amides. BASF. *BP* 731,008

3:4-Phthaloylisatoic acid anhydride—



is condensed with an amino compound having < 1 H attached to N to give, with loss of CO_2, a 1-aminoanthraquinone-2-carboxylic amide. Thus 3:4-phthaloylisatoic acid anhydride, prepared by treating 1-aminoanthraquinone-2-carboxylic acid in nitrobenzene at 140°c. with $COCl_2$, is heated at 70°c. for 5 hr. in aq. methylamine.

R. K. F.

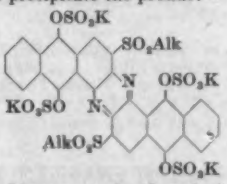
Chloromethylated Arylaminoanthraquinones and derived Water-soluble Onium Salts. General Aniline. *USP* 2,662,902

An α-arylamino-anthraquinone is treated with bis-chloromethyl ether, when CH_2Cl groups enter the aryl nucleus only. Thus 1-anilino-4-hydroxyanthraquinone is stirred with *bis*-chloromethyl ether in H_2SO_4 at 30–32°c. and the product separated by pouring into ice and water. *USP* 2,664,427

These chloromethyl derivatives are converted into *iso*-thiouronium and quaternary ammonium salts by treating with a thiourea and a tertiary nitrogen base respectively. The water soluble compounds thus obtained can be printed on cotton and used for colouring paper. Thus, the product of the above example is heated at 100°c. with aq. 1:1:3-trimethylthiourea, allowing the water to evaporate. A water-soluble bluish-violet derivative is obtained as a stiff paste. R. K. F.

Leuco - bis - 3 : 3′ - alkylsulphonyl - indanthrone Sulphuric Esters. American Cyanamid Co. *BP* 731,122

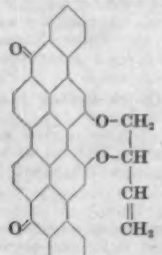
Leuco-2-acetylamino-3-alkyl (> 4 C) sulphonyl anthraquinone disulphuric ester (K salt) is heated in aq. NaOH until hydrolysis of the acetylamino group is complete; black nickel oxide, prepared from nickelous chloride is then added at 80°c. After concentrating by evaporation, KCl is added to precipitate the product—



which when dyed by conventional methods gives greenish blues faster to oxidising treatments than indanthrone itself. The starting material is made by first treating 2-acetylamino-3-bromo-anthraquinone at 90°c. with Na_2S and S in pyridine, alkylating the resulting Na 3-mercaptide with, e.g. dimethyl sulphate, and oxidising to the alkyl-sulphonyl derivative with $KMnO_4$ in acetic acid. Conversion to the leuco sulphuric ester follows by treating the product with chlorosulphonic acid in α-picoline in presence of Fe powder. R. K. F.

2:2′-Dihydroxydibenzanthrone Derivative— Vat Dye. DuP. *USP* 2,662,892

A 4-halogeno-1:2-butadiene is condensed with 2:2′-dihydroxydibenzanthrone or its alkali metal salt to give—



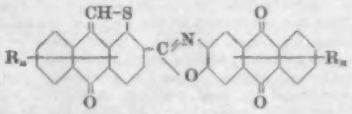
a bright green blue vat dye. Thus the mono Na salt of 2:2′-dihydroxydibenzanthrone ground to 60 mesh, K_2CO_3 and anhyd. sodium acetate are heated to 170°c. in *o*-dichlorobenzene. 4-Chloro-1:2-butadiene is added quickly and the whole refluxed for 18 hr. R. K. F.

Chloromethylated Pyranthrone, Anthanthrone and Dibenzpyrenequinone Vat Dyes. General Aniline. *USP* 2,666,767

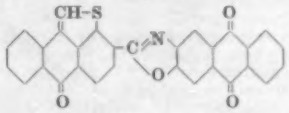
Pyranthrones, anthanthrones and dibenzpyrenequinones are treated with dichlorodimethyl ether to form chloromethyl derivatives, which are convertible to the corresponding water-soluble onium salts, and which produce the corresponding methyl derivative on the fibre after vatting. Thus dichlorodimethyl ether is added slowly to a solution of dibenzpyrenequinone in H_2SO_4 and the whole stirred at 75°c. for 4 days. The product is isolated by pouring into water. R. K. F.

Fluorescent Vat Dyes. General Aniline. *USP* 2,383,717

Dyes of formula—



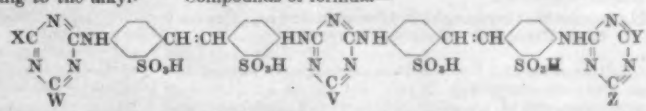
(R = any inert substituent, e.g. Alk, alkoxy or Hal; $n > 0$) yield greenish-yellow dyeings on cotton which give yellow fluorescence under ultra violet radiation. The dyeings are very fast to chlorine and washing. Thus nitrobenzene (100 parts by vol.), thionyl chloride (8·9) and 1:9-thiophenanthrone-2-carboxylic acid (7 by wt.) are stirred together at 75–80°c. for 90 min. The mixture is then refluxed so as to distil off 33 parts by vol. of liquid. 2-Amino-3-oxyanthraquinone (6 by wt.) is then added and the whole agitated under reflux at 200–205°c. for 4 hr., the mass cooled to room temperature, filtered, washed with nitrobenzene and then with benzene and dried. The product (5 by wt.) is stirred at 145°c. for 30 min. in conc. H_2SO_4 (50 by vol.), cooled and poured into water. The dye obtained is purified by boiling in aq. alkaline NaOCl. It has the formula—



C. O. C.

Fluorescent Brightening Agents derived from 4:4′-Diaminostilbene-2:2′-disulphonic Acid. Ciba. *BP* 732,139

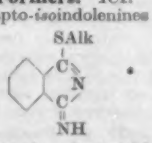
Compounds of formula—



(V, W, Z = NH_2 or residue of a primary or secondary amine attached by N to the triazine ring; X and Y = Cl, the residue of an aromatic hydroxy cpd. attached through –O– to the C atom of the triazine ring, or of an aliphatic or aromatic mercapto compound attached through –S– to the C atom of the triazine ring or = W or Z) are fluorescent brightening agents having affinity for cellulose. They are obtained by treating 2 mol. of a 4-aminostilbene-2:2′-disulphonic acid containing in the 4′-position a group convertible to an amino group, with 3 mol. of cyanuric chloride and 3–5 mol. of ammonia or of a primary or secondary anion compound, or with 2 mol. of an aromatic hydroxy or mercapto compound and 3 mol. of ammonium or a primary or secondary amino compound, the 4′-group being converted into an amino group at any suitable intermediate stage. C. O. C.

1 - Imino - 3 - alkylmercapto - *iso*indolenines—Phthalocyanine Formers. ICI. *BP* 731,257

1-Imino-3-alkylmercapto-*iso*indolenines of formula—



form a phthalocyanine *in situ* when padded or printed on a textile material in presence of a metal compound, e.g. a Cu or Mg salt, and later heated. They are made by first treating phthalonitrile at 30°c. with an alcoholic suspension of Na_2S until a test sample is soluble in water. The resulting imino-thiophthalimidine is separated by pouring into aq. $NaHCO_3$ and converted into the imino-

alkylmercapto*iso*indolenine by dissolving in aq. NaOH and alkylating by adding dimethyl sulphate at 20–25°C.
R. K. F.

Solvent-soluble Phthalocyanine Derivatives. DuP. *USP* 2,662,895

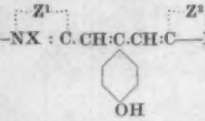
Phthalocyanines are treated with an organic hypochlorite R·O·Cl (R = *tert.*-butyl, *tert.*-amyl, 2-phenyl-*iso*propyl or 2-*p*-tolyl-*iso*propyl) to give red-brown products, soluble in, e.g. ethylene-glycol-mono-ethyl ether, which are converted to the original phthalocyanine by reducing, e.g. with $NaHSO_3$. They may be used to colour, e.g. cotton by padding from a solvent and treating with a reducing agent. Thus Cu phthalocyanine in its α-form, produced by acid pasting, is stirred with *tert.*-butyl hypochlorite in methanol at 25°C. for 70 min. The unchanged phthalocyanine is filtered off, and the filtrate allowed to stand, when brown crystals consisting of the addition product of Cu phthalocyanine (1 mol.) and *tert.*-butyl hypochlorite (1 mol.) are deposited.
USP 2,662,896

Products with similar properties are made by treating a phthalocyanine with methanol in presence of Cl_2 or Br_2. In addition to their solvent-soluble properties, they are characterised by reproducing the original phthalocyanine when treated with alcoholic ascorbic acid.
USP 2,662,897

Similar products result from the action of dibenzoyl and halogenated dibenzoyl peroxide on phthalocyanines.
R. K. F.

***meso*Phenylcarbocyanine Dyes.** Kodak. *BP* 733,397

Dyes of formula—



(R^1 and R^2 = same or different, subst. or unsubst. Alk; X = acid radical; Z^1 and Z^2 = atoms to complete a benzthiazole or naphthathiazole nucleus), e.g. 3:3′-diethyl-9-*p*-hydroxyphenylthiocarbocyanine iodide, are useful photographic sensitisers.
C. O. C.

Hydrophobic Organophilic Ultramarine and Peacock Blue Pigments. American Cyanamid Co. *USP* 2,683,702

Ultramarine and Peacock Blue pigments are formed into an aqueous slurry with 2–10% by wt. of a mixture or precondensate of an alkylated methylol melamine and *N*-alkylol fatty acid amide (chain of 10–20 C), e.g. dimethylated trimethylol melamine and stearamide, dried and baked. The product is extremely hydrophobic, satisfactorily organophilic and has good resistance to lithographic fountain solution. It can also be used in emulsion paints which dry to a glossy film.
C. O. C.

Blanc Fixe. Eastman Kodak Co. *USP* 2,685,499

When precipitating blanc fixe by adding barium chloride to an aqueous solution of a sulphate, dispersing the barium ion as rapidly as possible in the reaction mass, so that the content of sulphate ion in the reaction is in a greater equivalent amount than the barium ion over practically the entire zone of reaction, prevents any variation in colour of the product.
C. O. C.

Calcium Carbonate. J. Vasátko and V. Krizan. *BP* 733,418

The precipitate formed when sugar solutions are purified by treatment with lime and CO_2 is separated into different fractions by elutriation. Water is then removed by filtration or centrifuging from one of the middle fractions to yield purified calcium carbonate or a chalk like material. If this is dark coloured it can be bleached preferably with an oxidising agent or the original precipitate can be bleached before elutriation.
C. O. C.

Reducing the pH of Furnace Blacks. Phillips Petroleum Co. *USP* 2,682,448

The furnace black, either loose or pelleted, is subjected to controlled oxidation by a gas without causing appreciable loss from combustion.
C. O. C.

Simultaneously Pelleting and Rendering Furnace Black Non-scorching. Godfrey L. Cabot. *USP* 2,686,107

Ferro-organic Pigments. IC. *USP* 2,683,154

A slurry of ferric hydroxide is refluxed with a polymerised or oxidised and polymerised unsat. monocarboxylic acid or its ammonium salt, e.g. oxidised polymerised dehydrated castor oil fatty acid, using a ratio of > 1 acid-radical to three iron atoms. The products are not, or only very slightly, soluble in organic solvents, insoluble in water and used in organic coating compositions yielding translucent reddish-brown films.
C. O. C.

Extenders or Reinforcing Pigments. Northern Minerals. *USP* 2,686,731

Pigments of particle size 20–50 mμ. and of bulk density > 10 lb./c. ft. are produced by dissolving natural or synthetic calcium silicate in mineral acid to a clear solution. This solution is then diluted to precipitate an easily filterable hydrated silica whose particle size may be varied by varying the conditions of precipitation. Dilute slurries or solutions of Mg and/or Ca and/or Sr and/or Ba and/or Zn and/or Al compounds may be added during the dilution so as to produce mixed pigments.
C. O. C.

Dispersible Dye and Pigment Powders. General Aniline. *USP* 2,685,494

Marasperse CB, a modified or partly desulphonated ligno sulphonate obtained by the process of *USP* 2,371,136–7, is a good dispersing agent for use in producing powdered vat or other insoluble dyes or pigments, including inorganic pigments.
C. O. C.

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p-Nitrophenol as Sighting Colour for Wool and Non-cellulosic Synthetic Fibres (X p. 564).

Complexes of Eriochrome Black T with Calcium and Magnesium (XIV p. 567).

New Organic Reagent for Silver and Copper (XIV p. 567).

V—PAINTS; ENAMELS; INKS

PATENTS

Dye-toned Printing Inks. J. M. Huber Corpn. *BP* 732,194

Addition to printing ink of a solution of an organic dye which is insoluble in the ink vehicle enables considerable quantities of dye to be incorporated. When such inks are printed onto paper the vehicle is absorbed by the paper and the dye is precipitated apparently onto the surface of the carbon black. Such inks retain their toned effect permanently.
C. O. C.

Black Hectograph Ink. General Aniline. *USP* 2,684,908

Addition of a large proportion of Spirit Nigrosine (*C.I.*684) to the dyes which combine to give the black, results in an ink which yields a good black in many more copies without deterioration towards the hue of one of the component dyes. Apparently as the shading dyes become exhausted the Spirit Nigrosine prevents any of the shading dyes becoming dominant and so maintains the black throughout.
C. O. C.

High-reflectance Quick-drying Marking Inks. U.S. Secretary of the Navy. *USP* 2,684,303

Inks for marking on glass or clear solid plastic surfaces are made by dispersing TiO_2 in an aqueous solution of dextrin containing gum tragacanth, bentonite, and a wetting agent.
C. O. C.

Zein Ink Vehicle. Time. *USP* 2,684,909

Zein inks which are stable on the press rolls, insensitive to wide variations in R.H. and readily controlled as to drying temperature so that fast drying of the ink can be obtained e.g. at 90–165°C. contain as solvent a solvent for zein and a compound which alone is not a solvent for zein but becomes a solvent in the presence of water or a zein solvent, e.g. a mixture of ethylene glycol and a compound of formula $R^1·O·[CHR^2·CHR^3·O]_x·H$ (R^1 = Alk of 1–4 C; R^2 and R^3 = H or Alk of 1–4 C; x = 1, 2 or 3), e.g. butyl ether of diethylene glycol.
C. O. C.

Nickelous Oxide as a Colourless, Transparent Protective Agent in Organic Coatings. DuP. *USP* 2,683,668

Addition of precipitated nickelous oxide to organic coating compositions yields films which when > 0·005 in. thick possess the following properties —

(1) Absence of visible original colour from the nickelous oxide; (2) Absence of original opacity caused by the nickelous oxide; (3) Long life of the film due to presence of the nickelous oxide; (4) Absence throughout the life of the film of colour and/or opacity developed by the nickelous oxide. C. O. C.

Ink for Printing on Resinous Films. Sherwin-Williams Co. *USP* 2,686,736

An oil-in-water emulsion is used which contains as the pigment binder a latex whose nature corresponds very closely to that of the films it is desired to print, e.g. a vinyl chloride latex would be used for an ink to print on vinyl chloride–vinyl acetate copolymer sheeting. Very sharp prints of good adhesion are obtained. In addition the inks dry very quickly and there is no puckering of the plastic sheet being printed. C. O. C.

Aqueous Dispersions of Fluorochlorocarbon Polymers. M. W. Kellog Co. *USP* 2,686,767

Aqueous dispersions of resinous polymers of e.g. trifluorochloroethylene containing as melting agent an alcohol of < 9 C, ketone of < 7 C or a non-ionic substituted polyethylene oxide of < 31 C, are used as coating agents or impregnants followed by fusing or sintering at about 200°c.

USP 2,686,770

Powdered plasticised polytrifluorochloroethylene is dispersed in a glycol or its derivative, e.g. ethylene glycol or dioxane or 2-ethoxy-ethanol-1 diluted with xylene. The dispersion is used e.g. in dip coating, the coated articles being dried between each dip and finally baked at 350–400°c. for 1–5 min. C. O. C.

Ceramic Colour Compositions Applicable by Screen Printing. B. F. Drakenfeld & Co. *USP* 2,682,480

The pigment is dispersed in a vehicle comprising a wax and a resin solution, e.g. equal parts by weight of hydrogenated wood rosin, hydrogenated methyl ester of rosin and stearamide with if desired ethyl cellulose and a hard waxy polyethylene glycol. C. O. C.

Wood Stain containing a Fluorescent Brightening Agent. Chadeloid Corpn. *BP* 732,450

Application of an organic solvent solution of a fluorescent brightening agent brightens the finish, and brings out the grain of the wood. If desired a water-soluble dye can be incorporated in the solution. C. O. C.

VI— FIBRES; YARNS; FABRICS

Mechanical Electrostatic Charging of Fabrics for Air Filters. L. Silverman, E. W. Conners, and D. M. Anderson. *Ind. Eng. Chem.*, **47**, 952–960 (May 1955).

A mechanically induced electrostatic charge on certain fabrics is used to remove dust from air at room temp. Results are given of tests on a two-stage filter unit using a fixed fabric *A* charged to one sign by a moving fabric surface *B* as a charging stage, followed by a moving belt of *B*, charged by a moving fabric *A*, as the precipitation stage. W. K. R.

Electrokinetic Potentials of Textile Fibres. M. von Stackelberg, W. Kling, W. Benzel, and F. Wilke. *Kolloid-Z.*, **135**, 67–80 (Feb. 1954).

Zeta potentials are obtained from the rate of flow, under a potential difference, of an aqueous solution of electrolyte or surface-active solute through a plug of textile fibres. The potentials for all fibres in KCl solution are negative, increasing with the concn. of KCl to a max. at about 10^{-4}N-KCl and then decreasing to a small value at N-KCl. Potentials for acetate rayons in KCl soln. increase linearly with temperature. Anion-active Na alkyl sulphates increase potentials for hydrophobic fibres, but with hydrophilic fibres the increase begins suddenly when the solute concentration exceeds the critical micelle concentration. Cation-active dodecylpyridinium chloride reverses the charge on the fibres. A non-ionic compound of dodecyl alcohol and ethylene oxide reduces the potential in the presence of KCl. Methylcellulose and gelatin reduce the potential, especially if salts are present. Na cellulose-glycollate increases the negative potential greatly at concn. up to 0·01 wt. %, but above this the viscosity of the solution controls the flow rate. The magnitude of the charge on the fibre surface increases with increasing absorptive capacity of the fibre for surface-active agent and with decreasing water absorption. The effectiveness of ionic surface-active agents in washing is attributed to electrostatic repulsion of like charges on the fibre and on the soil. W. R. M.

Factors affecting the Fluorescence of Cotton. G. H. Lourigan. *Amer. Dyestuff Rep.*, **44**, P 348–P 349, P 359 (23 May 1955).

It is suggested that fluorescence depends on the amount of degraded cellulose associated with the tested samples; the materials which contribute to the fluorescence are water-soluble, as hot aqueous solutions decrease the effect, with the exception of treatments known to produce degradation. Fluorescence may be responsible for some dyeing troubles, following blending of cottons from different crops which fluoresce differently. J. W. B.

Crystalline–Amorphous Ratio of Keratin Fibres. I— Hailwood–Horrobin Analysis of the Water-adsorption Isotherms. C. H. Nicholls and J. B. Speakman. *J. Textile Inst.* **46**, T 424–T 426 (June 1955).

Water-adsorption isotherms of Lincoln, New Zealand Romney, and South African merino wools, mohair, and Lincoln wool esterified with methanol and HCl are determined at 25°c. The fraction accessible to water molecules, calculated by the Hailwood–Horrobin procedure, varies from 0·7 for Lincoln wool to 0·6 for mohair, but an anomalous result is obtained in that methylation apparently decreases the accessible fraction of Lincoln wool to 0·63. As it is unlikely that esterification increases crystallinity, it appears that the Hailwood–Horrobin analysis must not be relied on for quantitative estimation of the water-accessible fraction, though it may give the correct order of crystallinity of different keratins. The anomaly may be caused by methylation of free –COOH groups reducing their affinity for water and thus giving a high value for the inaccessible fraction. J. W. B.

Crystalline–Amorphous Ratio of Keratin Fibres. II— Hydrogen–Deuterium Exchange Reaction. R. W. Burley, C. H. Nicholls, and J. B. Speakman. *J. Textile Inst.*, **46**, T 427–T 432 (June 1955).

The proportion of amorphous material in wool is assessed by exposing a weighed sample to saturated deuterium oxide vapour and expressing the gain in dry weight, due to the hydrogen–deuterium exchange reaction, as a fraction of the maximum possible weight increase, calculated from the constitution as at present known on the assumption that it is all accessible. The order of accessibility of 3 wools and mohair is shown to be the same as that given by the Hailwood–Horrobin analysis of water-adsorption isotherms, but the exchange reaction gives much higher values of accessibility, ranging from 0·87 for Lincoln wool to 0·82 for mohair. The reaction is also used to reveal differences in accessibility of various types of fibre from New Zealand Lincoln hogget's wool, fibres from the first follicles to develop being most, and those from the last to develop being least, crystalline. J. W. B.

Chemical Constitution of some Silk Fibroins and its Bearing on Physical Properties. F. Lucas, J. T. B. Shaw, and S. G. Smith. *J. Textile Inst.*, **46**, T 440–T 452 (June 1955).

The amino acid composition of a number of silk fibres of different origin, and that of the Byssus fibre of *Pinna nobilis*, are determined by hydrolysis in 6N-HCl, treatment of the mixed amino acids with 1-fluoro-2:4-dinitrobenzene, separation of the dinitrophenylamino acids by column chromatography, and determination of the amount of each by estimation of ultraviolet absorption. The ratio of the amounts of long-side-chain to short-side-chain amino acids enables the silks to be divided into 3 main groups: the value, $\times$ 100, varies from 3·2 to 9·3 (mean 5·5) for the Anaphe group, varies from 22·1 to 30·0 (mean 26·6) for the Tussah group, and is 13·5 for *Bombyx mori*. For Byssus fibres it is 156. Load–extension characteristics are measured in air and in water and can be correlated with the distribution of amino acids: at 65% R.H. the Anaphe group show comparatively low extension to breaking point; *Bombyx mori* fibres show a steadily curved graph giving 24% extension at break; the Tussah

group shows a much greater extension; while the Byssus fibre gives an extension of 50% for a stress of little more than 1 g./denier. Elastic recovery is also studied. Moisture regains are measured and do not appear to differ greatly—presumably they depend more on the peptide links than on amino acid distribution. The apparent density of the fibres changes when measured in benzene and then in water, and the change is related to the ease with which water can penetrate the fibres and, therefore, to the proportion of amino acids with bulky side-chains. J. W. B.

Electrical Resistance of Fibrolane and Vicara. G. E. Cusick and J. W. S. Hearle. *J. Textile Inst.*, **46**, T 369–T 370 (May 1955).

Studies described earlier (J. W. S. Hearle, *J. Textile Inst.*, **44**, T 117 (1953)) are extended by experiments on Fibrolane and Vicara protein fibres. J. W. B.

Fibres from New Polymers. R. J. W. Reynolds. *J. Textile Inst.*, **46**, P 328–P 331 (May 1955).

A brief survey is given of fibre production from polymers and copolymers, with particular reference to the influence of composition, i.e. the nature and the arrangement of the monomer units, on fibre properties. J. W. B.

Melting Behaviour of Crystalline Polymer Fibres. T. R. White. *Nature*, **175**, 895–896 (21 May 1955).

Using differential thermal analysis in the study of drawn yarns of 6-, 6,6-, and 6,10-nylons, polyethylene terephthalate, certain copolyamides, and Perlon U, two distinct heat absorptions are found to occur in separate temperature regions. These two heat changes appear to accompany respectively the disorientation of crystalline regions and actual fusion of crystallites. The shape of the melting curve, at a fixed heating rate, depends on crystallinity and degree of orientation of crystallites. The melting curves are a valuable method of characterisation, and small differences in degree of orientation of highly oriented yarns are more readily detected than is possible by X-ray methods. W. R. M.

Crystal Structure of Polycaproamide (6-Nylon). D. R. Holmes, C. W. Bunn, and D. J. Smith. *J. Polymer Sci.*, **17**, 159–177 (June 1955).

The crystal structure of 6-nylon has been determined from X-ray diffraction results on drawn rolled fibres. 6-Nylon melts 50°c. lower than 6,6-nylon. The unit cell contains 8 chemical units and is monoclinic with a calculated density of 1·23. The observed density for a drawn monofilament is 1·16. The structure consists of planar chains of CH_2 groups and amide groups tilted 7° for the (001) plane. Alternate chains in the plane are oppositely directed, permitting all hydrogen bonds to be formed perfectly. The hydrogen-bonded sheets are packed in an "up-and-down" staggered configuration along the *c*-axis. From a general survey of polyamide m.p. it appears that the determining factor is the number of CH_2 groups between the amide "anchor points". Polymers with odd numbers of CH_2 groups melt at lower temperatures than those with even numbers. The lower m.p. of 6-nylon compared with 6,6-nylon is not due to deficient hydrogen bond formation and must be ascribed to some other cause, possibly connected with propagation of vibration along odd-number chain segments. W. R. M.

Species of Low Molecular Weight in Polycaproamide. C. W. Ayers and S. Smith. *Chem. and Ind.*, 684–685 (11 June 1955).

The product of polymerisation of caprolactam has been fractionated by solvent extraction and by ion-exchange resins. Fractions were analysed by electrophoresis, paper partition chromatography, and infrared techniques. Monomeric, dimeric, and trimeric caprolactam, aminocaproic acid, and higher linear oligomers of aminocaproic acid were detected, and in addition basic compounds with R_F values less than aminocaproic acid, acidic non-amino compounds, and a very sparingly soluble compound which did not absorb in the infrared. W. R. M.

Some Properties of Undrawn 6-Nylon Yarn. E. H. Boasson and H. J. H. Scheers. *J. Polymer Sci.*, **17**, 311–314 (June 1955).

The increase in length following water absorption after dry spinning has been studied. It increases with increasing birefringence of filaments, and at high values of the birefringence the increase in length is much greater than corresponds to the water uptake. Considerable increase in length occurs if the fibre is immersed in water immediately after spinning. This, and the rate of increase of length, increase with the birefringence of the filament and the temperature of the water. Much of this increase in length is reversible. If birefringence is determined immediately after spinning, an inhomogeneous birefringence is observed which is ascribed to diffusion of water from the immersion liquid. W. R. M.

PATENTS

Ramie Fibres. J. Rinse. *USP* 2,687,351

Ramie fibres are recovered from the plant with little loss in strength by treating the raw material with hot aqueous alkali metal hydroxide (about 3–15% calculated as NaOH) containing 0·5% ZnO. This gelatinises but does not remove the binding substances which are removed by washing with water at 70°c. followed by souring with dilute acid. C. O. C.

Viscose Rayon Free from Black Specks or Stains. DuP. *USP* 2,686,104

Incorporation into the acid salt coagulating bath of 0·01–0·1% of the weight of the liquor of salicylic acid, salicylaldehyde, salicylal ethanolamine, disalicylal ethylene diamine, disalicylal propylene diamine, dimethyl glyoxime, diacetone alcohol, α-benzoin oxime or ammonium nitroso-β-phenylhydroxylamine prevents formation of specks or stains due to traces of lead in the bath. C. O. C.

Regenerated Cellulose having Affinity for Acid Dyes. DuP. *USP* 2,686,103

Incorporation in the viscose solution of a styrene-maleic anhydride interpolymer or a derivative imparts to the filaments spun from it affinity for wool dyes, water-repellency, improved handle, resiliency and insolubility in the usual cellulose solvents. C. O. C.

Viscose Rayon of Decreased Water Absorption. American Cyanamid Co. *USP* 2,684,343

Addition of an alkylated polymethylol melamine resin containing > 2 methyleneoxy groups and > 9 C when there are < 4 methylene oxy groups to the spinning solution yields filaments of decreased water absorptive properties. C. O. C.

Animalising Cellulose with *iso*Melamine Resins. American Cyanamid Co. *USP* 2,684,920

Incorporation into cellulose of non-colloidal aminotriazine resins, e.g. that obtained from butyl *iso*melamine hydrochloride (109 parts) and 37% aq. HCHO (122) brought to pH 7·1 with triethanolamine, imparts affinity for acid dyes and markedly decrease the water absorptive properties. They may be applied by impregnating the fibre with the precondensate and then baking. C. O. C.

Improving the Properties of Polyvinyl Alcohol Fibres, Films, etc. Kurashiki Rayon Co. *BP* 732,934

The dry and wet heat resistance and elastic recovery properties of polyvinyl alcohol fibres are much improved by treatment with dialdehydes, keto aldehydes or their acetals, e.g. glyoxal, succinnic aldehyde, terephthalic or 2:5-dichloro terephthalic aldehyde or their acetates. The treatment is preferably applied to fibres whose softening temperature in water has been raised to > 80°c. by heating, e.g. for 3 min. at 215°c. in superheated steam. C. O. C.

Improving Polyvinyl Alcohol Fibres. Kurashiki Rayon Co. *BP* 733,102

The dyeing and mechanical properties of polyvinyl alcohol fibres which shrink $< 5\%$ in water at 80°c. are improved by treating with an amino-opd. such as a *sec.*-aminoaldehyde, *sec.*-aminoacetals, *tert.*-aminoaldehyde and a *tert.*-aminoacetal. W. G. C.

Delustred Polyamide Fibres. Vereinigte Glanzstoff-Fabriken. *BP* 733,007

Delustred polyamide fibres having good stability to light are obtained by incorporating polystyrene in a fine dispersion prior to the spinning process. W. G. C.

Polyacrylonitrile Fibres. Eastman Kodak Co. *USP* 2,682,518

Fibres composed of polyacrylonitrile (60–90 parts by wt.) and an *iso*propenyl acetate–low alkyl acrylate copolymer (40–10) readily dye deeply with disperse dyes.

USP 2,682,519

Introducing free hydroxyl groups into interpolymers of vinyl acetate and unsaturated esters such as methyl acrylate, dimethylmaleate or *iso*propenylacetate and then mixing the interpolymers with acrylonitrile polymers of 75–100% acrylonitrile content, produces a composition which can be spun into fibres which dye deeply with vat, direct, disperse, basic and acid dyes. C. O. C.

Spinning Polyacrylonitrile Fibres. Courtaulds. *BP* 732,135

Threads obtained by coagulation of aqueous salt solutions of polyacrylonitrile are easier to stretch when the coagulating bath contains > 5% of the water-solution salt, e.g. calcium thiocyanate and a water-miscible alcohol, e.g. methanol. W. G. C.

Readily Dyed Polyacrylonitrile. DuP. *USP* 2,686,136

An aqueous polyacrylonitrile gel, e.g. as film or fibre, treated with an aqueous solution of a formaldehyde resin precondensate and then dried is readily dyed with acid, disperse, direct and vat dyes. C. O. C.

Solvent for Polyacrylonitrile (III p. 552).

2-Sulphoalkyl Ethers of Cellulose—Textiles having Ion-exchange Properties, Organdie Finish (X p. 565).

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Energetics of Washing Processes for Oil Stains. W. Kling and H. Lange. *Kolloid-Z.*, **142**, 1–5 (June 1955).

Kling and Koppe's calculations (*Melliand Textilber.*, **30**, 23 (1949)) for the amount of work in the washing process are extended to the case of an oil stain not spontaneously removed by the detergent but only partly wetted to a definite equilibrium contact angle. An equation is derived for the additional work involved for complete removal, and this residual work in two series of measurements for the respective systems glass and Perlon with paraffin oil and sodium dodecyl sulphate is shown to be decreased by an active anionic detergent. H. H. H.

Jute Wax—Isolation, Properties, and Chemical Composition. W. G. Macmillan, A. B. Sen Gupta, and A. Roy. *J. Indian Chem. Soc.*, **32**, 79–84 (Feb. 1955).

The apparent wax content of jute fibres (determined by extraction with 1 : 2 alcohol–benzene) is 0·5–0·6%, representing only 18% of the waxy components of the bast. However, the alcohol–benzene extract was shown to contain about 60% of resinous impurities. The purified wax has m.p. 58–62°c. and consists of fatty acids (33%), alcohols (47·3%), sterols (7·1%), hydrocarbons (9·7%), and inert matter (2·9%). The contents of saturated and unsaturated acids are 29·1% and 3·9% respectively. A. J.

Peroxide Bleaching. B. P. Ridge. *J. Textile Inst.*, **46**, T 371–T 374 (May 1955).

Reasons are presented to explain the American preference for peroxide bleaching in contrast to the British use of hypochlorite. J. W. B.

Single-stage Continuous Peroxide Bleaching. T. E. Bell, H. D. Terhune, H. L. Potter, and M. H. Rowe. *Amer. Dyestuff Rep.*, **44**, P 317–P 319, P 327 (9 May 1955).

The single-stage continuous peroxide bleaching process as applied to cotton knit goods, yarn-dyed piece goods, and grey piece goods is discussed, and comparisons are made between equipment and running costs of single- and multi-stage processes. Apart from being more economical, the single-stage process results in smaller weight losses, less width shrinkage, and a fuller handle. J. W. B.

Fluorescent Brightening Agents in Wool Bleaching. R. Keppler. *Textil-Praxis*, **10**, 385–387 (April 1955).

Recipes are given for wool bleaching by oxidation and reduction methods including the incorporation of fluorescent brightening agents. B. K.

PATENT

Stripping Colour from Keratinous Material. Harris Research Laboratories. *USP* 2,685,496

The material is treated with a reducing agent for keratin in presence of an alkylene dihalide as described in *USP* 2,508,714 (J.S.D.C., **66**, 660 (1950)) and is then treated either in the same or in a fresh bath with a different stripping agent or to different stripping conditions at lower pH than was used for the first treatment. The stripped materials have greater resistance to attack by oxidising and reducing agents, acids and alkalis, and a lighter colour than those stripped by hitherto-known processes. C. O. C.

Electrokinetic Potentials of Textile Fibres (VI p. 559).

Colouring and Wet Processing of Fabrics containing Fibrolane Regenerated Protein Fibre (VIII p. 562).

VIII— DYEING

Thermodynamics of Absorption by Fibres from Multicomponent Baths such as Dyebaths. H. J. White. *J. Physical Chem.*, **59**, 528–531 (June 1955).

Absorption by fibres from multicomponent baths is discussed using a strictly thermodynamic approach. Equations are given which can be used to determine the equilibrium constant for any distribution equilibrium in the system in terms of measurable quantities and of conventions adopted in defining activity coefficients. Other equations are given for free energy, and hence heat and entropy, changes for certain well defined processes which describe absorption. W. R. M.

The Barotor in Practical Application. F. S. Richardson. *Amer. Dyestuff Rep.*, **44**, P 360–P 361, P 366 (23 May 1955).

A report is presented on the actual operation, under full load, of a Barotor (DuP) machine; details are given of mechanical and operational improvements. J. W. B.

Developments in High-temperature Dyeing. E. Vogel and M. Peter. *Textil-Rund.*, **10**, 175–178 (April 1955).

The requirements of high-temperature dyeing apparatus are considered with particular reference to the design of the Mono-Ferma and Bi-Ferma models of Scholl AG, Zofingen. The dyeing of cellulosic fibres and of wool is briefly discussed, and a list of 53 light-fast, substantive Chlorantine (Ciba) dyes suitable for the high-temperature dyeing of native and regenerated cellulosic fibres is given. J. C. F.

Application of a Method of Integration in Dyeing at High Temperatures. L. Drijvers. *Dyer*, **113**, 695–701 (29 April 1955).

Translation of paper already abstracted (J.S.D.C., **70**, 470 (1954)).

Spun-dyed Fibres. J. V. Lund. *Amer. Dyestuff Rep.*, **44**, P 320–P 322 (9 May 1955).

A brief survey of their advantages, limitations, and the techniques of use. J. W. B.

Fixation of Direct Cotton Dyes. R. Gill. J.S.D.C., **71**, 380–389 (July 1955).

The various types of compounds used in the fixation of direct cotton dyes on cellulosic fibres are classified according to constitution, and examples are quoted to show the general structural requirements for fixation. A theory is presented to account for the fact that, besides increasing the fastness to wet processing, fixatives in general also may cause change in colour and decreased light fastness.

Unlike the older idea of precipitation within the fibre, this theory enables the effect of the fixative on the dichroism of the dyed fibres to be predicted. Results are presented to show that the resultant effects are as expected, and finally a comparison is drawn between direct dyeings

aftertreated with cationic fixatives and acid dyes on wool. The conclusion is drawn that there is a definite limit to the increase in fastness to washing with soap, or other anionic detergents, which may be attained with cationic fixatives generally. AUTHOR

Dyeing Jute Yarn. Anon. *Dyer*, **113**, 581–582 (15 April), 659–661 (29 April), 821–823 (27 May), 901–902 (10 June), 983–985 (24 June 1955).

A general practical survey of the dyeing of jute yarns with direct, acid, and basic dyes, including descriptions of the types of machines used for this purpose. A. H.

Electrophoretic Study of Bovine Plasma Albumin–Methyl Orange Complex at 25°C. M. A. Cohly. *J. Indian Inst. Science*, **37 A**, 89–97 (April 1955).

The interaction between bovine plasma albumin ($1{\cdot}49 \times 10^{-4}$M.) and Methyl Orange ($1{\cdot}53-25{\cdot}0 \times 10^{-4}$M.) at 25°C. in presence of an acetate buffer at pH 5·57 has been studied by electrophoresis. The dissociation constant of the protein–dye complex was found to be $3{\cdot}6 \times 10^{-4}$, and the maximum number of dye molecules combined per molecule of protein was 20. Contrary to previous reports in the literature, the electrophoretic patterns did not show any asymmetry in the complex boundary at higher dye concentrations. W. K. R.

George William Oldham, Silk Dyer, Moll Spring Dyeworks, Netherton, near Huddersfield. A. Thomson. J.S.D.C., **71**, 399–401 (July 1955).

Colouring and Wet Processing of Fabrics containing Fibrolane Regenerated Protein Fibre. R. C. Cheetham. *Dyer*, **113**, 741–746 (13 May 1955).

Instructions are given for dyeing and finishing fabric blends of Fibrolane protein fibre with a variety of other fibres. In order to reduce swelling to a minimum and retain the greatest strength of the Fibrolane, mildly acid conditions (pH 4–6) should be aimed at. A sodium acetate–acetic acid buffer is used when desizing and scouring with, respectively, a proprietary destarching agent and a synthetic detergent (preferably non-ionic). Bleaching is not necessary unless required by other components in the blended fabric, in which case 2-vol. H_2O_2 + pyrophosphate are used at pH 8, followed by an acetic acid rinse. As alternatives H_2O_2 with an acid stabiliser or $NaClO_2$ + 3–4% acetic acid can be used. Fluorescent brightening agents are sometimes resorted to, but never hypochlorites. Optimum pH of the dyebath for Fibrolane is 4, but for effective dyeing of the Fibro in a Fibrolane–Fibro blend with direct dyes pH 6 must be used, this being the minimum at which cellulose fibres are successfully dyed. Many aggregated acid dyes also are satisfactorily dyed at this value. The effects of salt concn., temp., and time are discussed, and methods described for producing solid shades, reserve, and multicoloured effects. Selection of dyes, aftertreatments, crease-resist finishes, and drying equipment are the subjects of further discussion, and the correction of unlevel dyeings is described. If stripping is necessary, neutral hydrosulphite or Formosul–formic acid can be used, but not hypochlorites. An appendix gives four tables of direct dyes—(1) dyes at pH 6 to dye Fibro and reserve Fibrolane, (2) dyes giving solid shades on Fibro–Fibrolane at pH 6, (3) dyes giving deeper colours on Fibrolane than on Fibro at pH 6, (4) dyes producing tone differences on Fibrolane and Fibro at pH 6. A. H.

Dyeing Orlon Acrylic Fibre and Blends with Other Fibres. J. F. Laucius, R. A. Clarke, and J. A. Brooks. *Amer. Dyestuff Rep.*, **44**, P 362–P 366 (23 May 1955).

Recipes are given for dyeing Orlon with disperse, acid, basic, and vat dyes, and for dyeing blends of Orlon with wool, rayon, cotton, and nylon. J. W. B.

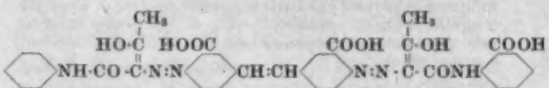
Acrilan Dyeing. W. H. Hindle. *Amer. Dyestuff Rep.*, **44**, P 323–P 327 (9 May 1955).

Recipes are given for dyeing Acrilan, and blends of Acrilan with acetate rayon, viscose and acetate rayons, wool, and nylon, with direct, disperse, chrome, vat, leuco vat ester, level- and neutral-dyeing acid, and neutral-dyeing metal-complex acid dyes. J. W. B.

PATENTS

Dyeing or Printing Cellulosic Textiles with Metal-complex Azo Dyes. Ciba. *BP* 732,925

Cellulosic textiles are readily dyed or printed by using a neutral to alkaline liquor or paste containing (a) a solid amine containing at least one amino group separated by 2 C atoms from a hydroxy group, (b) a derivative of orthophosphoric acid and (c) a metal complex of a dye containing > 1 azo linkage of which at least one is present in an *o:o'*-dihydroxy azo group or an *o*-hydroxy-*o'*-carboxy azo group, the metal having atomic number 27–29. If desired instead of (c) there may be used a metal-free dye containing > 1 azo linkage of which at least one is in an *o:o'*-dihydroxy azo group, and an alkali-resistant compound of a metal of atomic number 27–29 capable of forming a complex with the dye. Thus the copper complex of the dye—



(5 parts), trisodium phosphate (3 parts) and 1:2-di(β-oxyethylamino)-ethane (2 parts) are mixed together. The mixture is then used to dye cotton using a liquor ratio of 30:1. The cotton is entered at 60°C. the temperature raised over 20 min. to 90–95°C., 10 min. after reaching this temperature 30% (on wt. of goods) of crystalline Na_2SO_4 is added and dyeing continued for 30 min. A brownish orange of good fastness to washing and light is obtained. C. O. C.

High-temperature Vat Dyeing using Trialkanolamines. American Cyanamid Co. *USP* 2,683,645

When applying vat dyes at high temperatures addition of > 1% of a trialkanolamine, e.g. triethanolamine, on the weight of hydrosulphite markedly reduces the rate of consumption of hydrosulphite.

USP 2,683,646

Manganese compounds, e.g. manganous chloride, have the same effect, much less than the stoichiometrical equivalent of that necessary to form manganese hydrosulphite being effective. C. O. C.

Dyeing Textiles with Vat or Sulphur Dyes. E. Haslam. *BP* 733,065

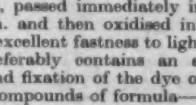
Bringing a textile impregnated with a vat or sulphur dye and a reducing agent into an atmosphere consisting wholly of hydrogen assists the action of the reducing agent. C. O. C.

Dye Emulsions for Dyeing and Printing of Textiles. S.A.I.C.A. Società Anonima Italiana Colori Affini. *BP* 733,099

Emulsions which penetrate the fibres without forming a coating on them consist of an aqueous dispersion or solution of vat or sulphur dyes emulsified in a liquid hydrocarbon. They contain 0·3–15·0% by weight of emulsifier, 5–25% of liquid hydrocarbon and no film-forming or polymerisable substance. Thus Indocarbon CL powder (*C.I.*1063) (6 parts by wt.) is mixed with a condensate of 1 mol. of amyl alcohol with several mol. of ethylene oxide (3) and an ethylene oxide-di*iso*hexylheptylphenol condensate (2). The paste so obtained is diluted with water (75) and poured into a mixture of aromatic hydrocarbons of boiling range 130–200°C. (14). The resulting emulsion may be applied directly to textiles by any convenient method. C. O. C.

Acid Vat Dyeing of Animal Fibres. General. *USP* 2,681,845

The materials are padded with an acid dispersion of a leuco vat dye, passed immediately into boiling aqueous acid for 1 min. and then oxidised in air. The resultant dyeings have excellent fastness to light and washing. The dispersion preferably contains an agent to accelerate penetration and fixation of the dye on the materials, for this purpose compounds of formula—



(R = aliphatic hydrocarbon of 10–18 C; m and n = same or different, each < 2 and together = 4–20), e.g. 8–10 mol. of ethylene oxide condensed with 1 mol. of oleyl amine. C. O. C.

Vat-dyeing Polyacrylonitrile Fibres. Deering Milliken Research Trust. *USP* 2,683,644

Level medium to deep dyeings are obtained by padding the reduced dye on to the material, and then passing it through a reducing bath containing about 30% Na_2CO_3 and finally oxidising and soaping. C. O. C.

Nature of Adsorption on Cellulose and Chitin from Aqueous Solutions (XI p. 565).

IX— PRINTING

Textile Printing in London and the Home Counties. D. King. J.S.D.C., **71**, 374–380 (July 1955).

A historical survey of the development of textile printing in the London area, from the Middle Ages until the present day. AUTHOR

Thickeners from Silicic Acid Gels. N. E. Fedorova and A. N. Burlakova. *Tekstil. prom.*, **14**, 33–35 (Oct. 1954): *Textil-Praxis*, **10**, 381–383 (April 1955).

Recipes are given for the preparation of textile-printing thickeners from silicic acid gels, and also methods for their subsequent removal from the cloth after printing. B. K.

Printing and Padding of Polyacrylonitrile Fabrics by the Sandocryl Printing Process. W. Flügel, A. Dürst, and E. Metzger. *Textil-Rund.*, **10**, 172–175 (April 1955).

The Sandocryl dyeing process, which employs dyes of the Sandocryl (S) range together with $CuSO_4$–Cu metal in place of the customary $CuSO_4$–conventional reducing agent, has been adapted to the printing of polyacrylonitrile fabrics. The printing paste contains finely divided Cu_2O, which undergoes disproportionation in presence of acid produced on steaming the prints—

$$Cu_2O \rightarrow Cu^{\circ} + Cu^{++} \rightleftharpoons 2\,Cu^{+}$$

The process is applicable to printing by screen, roller, Vigoureux, and block methods. The resulting prints possess excellent light and wet fastness. J. C. F.

PATENTS

Printing Screens. Eastman Kodak Co. *USP* 2,685,510

In making printing screens, use as the sensitive element of a double-coated silver halide emulsion layer, the outer emulsion being about half the thickness of the inner emulsion and containing yellow colloidal silver or other non-wandering yellow dye or pigment dispersed in it, enables longer times of exposure to be used without causing the tanned gelatin to be too hard. C. O. C.

αα′-Dichloroadipic Acid and Derivatives for Use in Azo Dye Printing Pastes. CFM. *BP* 732,379

Addition of salts of αα′-dichloroadipic acid to printing pastes containing a diazoamino compound in which the residue not taking part in dye formation is water-soluble and an azo coupling component imparts to the pastes outstanding stability at room temperature and after neutral steaming yields prints of intense and pure colour. Thus an equimolar mixture (80 g.) of 1-acetoacetylamino-4-chloro-2:5-dimethoxybenzene and the diazoamino compound of diazotised 5-chloro-2-amino-1-methylbenzene and the Na salt of methylaminoethane sulphonic acid is pasted with urea (50), ethanol (30), NaOH 38°Bé (30) and hot water (140). To the paste is added neutral wheat starch tragacanth thickener (500) in which αα′-dichloroadipic acid and NaOH 38°Bé (90) have been dissolved. Prints produced with this paste develop with neutral steam in 5 min. to a clear intense yellow. C. O. C.

Fabric Printing. G. J. L. Wooldrik. *BP* 732,421

The design is printed on paper with a fatty printing ink and is then transferred to the fabric which has been treated with a solution or emulsion of a synthetic resin but without filling the interstices of the fabric. The process is very suitable for reproducing pictures on fabrics. C. O. C.

Colouring Camouflage Fabrics. Stabilimenti di Ponte Lambro. *BP* 733,094

Fabrics which resist detection when viewed directly or indirectly by ordinary light and by infra-red radiation are produced by colouring different areas of them with different combinations of colouring matters. Each composition is formed of a mixture of elementary colouring materials, e.g. Phthalocyanine Green (40 parts), Dianisidine Orange (55), and Chrome Yellow (18·75) to produce a yellowish orange, the proportions being selected so that the mixtures used have all the same degree of absorption of infra-red rays. C. O. C.

Combined Embossing and Printing. Heberlein & Co. *BP* 732,417

A machine for embossing and printing fabric comprises a back-up roll for supporting the fabric, an embossing roll whose raised surfaces press against the fabric as it passes between the embossing and back-up rolls and a printing roll having raised surfaces identical with those of the embossing roll. After the cloth has left the nip formed by the embossing and back-up rolls, it passes through the nip formed by the printing and the back-up rolls so that the raised surfaces of the printing roll fit into the depressed portions of the fabric and print them. C. O. C.

Permanent Local Mechanical Effects on Crêpe Fabrics. Heberlein & Co. *BP* 732,210

Crêpe fabric, particularly rayon crêpe fabric, is impregnated with a softener, e.g. Ceranin F (8), dried while stretched, locally printed with a synthetic resin precondensate, passed through a friction or embossing calender, baked, washed and dried. There is no serious deterioration in strength of any part of the fabric. C. O. C.

Applying Transfers to Textiles. G. J. L. Wooldrik. *USP* 2,685,549

Improved results are obtained if a transfer printed with an organic pigment dispersed in a fatty vehicle is applied to a textile with the application of heat and pressure, the textile having been previously impregnated with an aqueous solution of a resinous precondensate and of a penetrating agent for the pigment, e.g. an aqueous solution containing 40% of thiodiethyleneglycol and 30% of polymethacrylic acid ester. C. O. C.

Decorated Moulded Thermosetting Synthetic Resins. Fairey Aviation Co. *BP* 732,774

The design is printed on to paper and the printed paper degreased, e.g. by treatment with a solvent. The paper is then impregnated with a solution of a synthetic resin, applied to the surface of the synthetic resin before curing is completed and then curing completed. C. O. C.

Colour Photography. ICI. *BP* 732,691

Manufacture and use of emulsion capable of recording an extended brightness range is described. C. O. C.

Colour Photography. E. Gretener. *BP* 733,446

A subtractive process which gives as good colour rendering as the additive processes. C. O. C.

Diazotype Printing Materials. Chemische Fabriek L. van der Grinten. *BP* 732,424

The light-sensitive surface contains a diazo compound and a polymer of a methacrylic ester applied before or together with the diazo compound. The polymer is applied as an aqueous dispersion and should be such as to yield a film of softening temperature > 60°c. Such materials have a non-glossy surface upon which it is easy to write with lead pencil. The copies show much improved image strength and greater uniformity of dye distribution in the under-exposed parts of the copy. C. O. C.

Reproductions and Printing Plates by Use of Diazo Compounds. Kalle & Co. *BP* 732,544

A plate having a light sensitive layer containing a water-insoluble 2-diazo-phenol-(1) or 1-diazo phenol-(2) of the benzene or naphthalene series is, after exposure to a light image, developed with an organic solvent or solvents in absence or presence of water and/or a salt. C. O. C.

Printing Multilayer Colour Cinema Film. Loew's. *BP* 732,942

An improved light-modulating strip enabling colour corrections to be effected while the film is passing continuously through a commercially available printing machine at a high rate of production. C. O. C.

Fusing Xerographic Images. U.S. Secretary of the Army. *USP* 2,684,301

Device for fixing xerographic powder images on paper using the vapour solvent condensation principle.

C. O. C.

Dyeing or Printing Cellulosic Textiles with Metal-complex Azo Dyes (VIII p. 562).

Dye Emulsions for Dyeing and Printing of Textiles (VIII p. 562).

X—SIZING AND FINISHING

Tetrakishydroxymethylphosphonium Chloride (T.H.P.C.) Flame-retardant Process on Cotton Fabrics. J. D. Guthrie, G. L. Drake, and W. Reeves. *Amer. Dyestuff Rep.*, **44**, P 328–P 332 (9 May 1955).

Tetrakishydroxymethylphosphonium chloride $(HOCH_2)_4PCl$, abbreviated T.H.P.C., is made by reaction of phosphine with H·CHO and HCl. It is applied by padding to e.g. cotton twill according to the following recipe: T.H.P.C. (1868 g.) is dissolved in 1800 g. water, to which triethanolamine (338 g.) is added. Trimethylolmelamine (1069 g.) and urea (1114 g.) are dissolved in 5062 g. of water, the two solutions are mixed, and they are applied to the cotton to give a 71·5% wet pick-up. After drying, curing, washing, drying, and softening, a fabric is obtained which contains 16·6% resin, fast to repeated laundering or dry cleaning. It shows very good fire-retardant properties and also has considerable wrinkle- and rot-resistance. The functions of the different components of the preparation are discussed—the T.H.P.C. furnishes a cross-linked condensation-type polymer with the melamine and imparts the flame resistance; the urea combines with liberated HCl and thus prevents tendering; and the triethanolamine stabilises the resin-forming solution at room temperature. J. W. B.

Flameproofing Compounds for Nylon. M. Fishman. *Amer. Dyestuff Rep.*, **44**, P 403–P 404 (6 June 1955).

Although pure nylon is flame-resistant, nylon in the presence of other fibres or finishing agents which form a framework for it can become inflammable. A suitable wash-fast flame-proof finish can be produced by thiourea–formaldehyde co-condensed with urea–formaldehyde.

J. W. B.

Reaction between Wool and Phenyl *iso*Cyanate. A. J. Farnworth. *Biochem. J.*, **59**, 529–533 (April 1955).

It has been shown that very little reaction occurs between wool and phenyl *iso*cyanate in aqueous media. Under anhydrous conditions considerable reaction occurs. Amino, carboxyl, and thiol groups all react, and probably phenolic, amide, guanidino, and glyoxaline groups also undergo reaction. The rate of reaction increases greatly with increasing temperature, but at higher temperatures protein degradation occurs simultaneously. Acetylation of wool with boiling acetic anhydride leads to reaction with a wide variety of side-chains. All groups capable of reacting with phenyl *iso*cyanate under anhydrous conditions probably react also with boiling acetic anhydride. Wool treated with phenyl *iso*cyanate exhibits a greatly reduced chemical reactivity, becoming more resistant to attack by acids, alkalis, and reducing agents. P. G. M.

Finishing Fabrics of Man-made Fibres. J. H. Andreen and L. D. Swan. *Amer. Dyestuff Rep.*, **44**, P 350–P 354 (23 May 1955).

New finishing techniques are described whereby the properties of new synthetic fibres may be brought out to their greatest advantage, e.g. wire-brushing of Terylene fabrics to prevent pilling, probably by distorting and locking the surface fibres into the fabric so that they are less free to work out. J. W. B.

PATENTS

***p*-Nitrophenol as Sighting Colour for Wool and Non-cellulosic Synthetic Fibres.** Milliken Research Trust. *USP* 2,683,647

Alkaline aqueous solutions of *p*-nitrophenol stain wool, nylon, etc. a bright yellow which though very stable is, even after crabbing or steam setting, readily removed by soap scouring followed by treatment with an acid. Normally a 0·3% solution of the Na salt is used.

C. O. C.

Sizing Nylon Yarns. Monsanto. *USP* 2,686,137

Nylon warps are sized with an aqueous solution at pH 2–5 of equimolecular proportions of vinyl acetate and maleic acid or anhydride or an alkyl half ester of maleic acid (Alk of 1–4 C). This gives excellent protection to the warps during weaving and the size is readily removed by simple scouring. C. O. C.

Preparation of Water-absorptive Unwoven Cellulosic Material. Lilia. *BP* 732,164

Water-absorptive unwoven cellulosic material, in web or pad form, e.g. surgical dressings, is treated with water-soluble chlorophyll either throughout or on one side only, e.g. by a spray. This can act as a barrier, as the absorptive capacity is reduced on that side; if used throughout it serves as an effective deodorant without appreciably reducing the absorptive properties. J. W. B.

Absorbent Cellulosic Products. American Cyanamid *USP* 2,683,087

Condensates of about 2·5–75·0 mol. of ethylene oxide per mol. of octadecylamine have affinity for cellulose to which they impart greatly improved absorbency.

USP 2,683,088–9

An aqueous dispersion of an alkylene oxide adduct of a hydroxyalkyl amide of a carboxylic acid of 14–22 C is added to the stock to produce a bibulous paper.

C. O. C.

Non-slip Finish. Monsanto. *USP* 2,701,218

Textiles made from fibres containing imido groups, e.g. nylon, are treated in a dilute acidic aqueous solution of colloidal silica until the desired amount of silica has been absorbed by the fibres. This imparts an antislip finish without affecting the handle. C. O. C.

Water-repellent Finish. FBy. *BP* 732,126

Textile or other sheet material is treated with a wax and/or liquid or solid hydrocarbon together with an aliphatic *iso*cyanate having a chain of > 9 C which may be mixed with an amine or polyamine or a mixture of a poly*iso*cyanate having a chain of < 10 C with an amine or polyamine having a chain of > 9 C. It is then treated with an aqueous solution of an Al or Zr compound at pH 3·0–5·5 and heated. C. O. C.

Crease Shedding and Dimensionally Stable Finish. De Bataafsche Petroleum MS. *BP* 732,573

Materials made from any type of fibre are impregnated with a glycidyl ether of a polyhydric alcohol containing on the average > 1 epoxy group per molecule and/or a polymer of a 2-alkenylglycidyl ether and then baked. Suitable compounds include the glycidyl ethers obtained by condensing a polyhydric alcohol with epichlorohydrin or polymers of allyl glycidyl ether of mol. wt. 300–600.

C. O. C.

Durable Wash-resistant Mechanical Finish and/or Design. Monsanto. *BP* 732,851

The fabric is impregnated with an aqueous solution of (1) a water-soluble polymer containing several carboxylic or carboxylic anhydride groups, e.g. a copolymer of vinyl acetate and maleic anhydride, and (2) a water-soluble or water-dispersible polymer containing > 2 hydroxyl groups, e.g. polyvinyl alcohol. It is then partly dried, given a mechanical finish and simultaneously or subsequently dried and baked. C. O. C.

Rendering Cotton More Resistant to Heat and Mildew by Treatment with Acrylonitrile. Institute of Textile Technology. *BP* 732,779

Cotton which is cyanoethylated so that it contains 0·1–0·6 cyanoethyl groups per anhydro-glucose unit is rendered resistant to heat and attack by mildew.

C. O. C.

Napping Tubular Knitted Fabrics. Proctor & Schwartz. *USP* 2,684,519

The fabric coming from wet processing in which it has been considerably stretched is dried, napped, dry normalised, e.g. treated by the process of *U.S.P.* 2,597,530 J.S.D.C., **69**, 31 (1953)), its moisture content altered either by moistening it or making it bone dry, and finally folding or batching it. If the fabric has not been much stretched during wet processing then the procedure is to wet normalise, e.g. by the above mentioned process, dry in absence of tension, nap, agitate and fold or batch. This yields a fabric which is dimensionally stable and of good handle. C. O. C.

New Threads based on Poylvinyl Derivatives. Société Rhodiacéta. *BP* 732,392

Multifilamentary polyvinyl derivative threads are given a woollen appearance by supertwisting, receiving the supertwisted thread under tension on a support, heat stabilising it, untwisting back nearly to normal, and heating again under tension sufficiently weak to cause spiral deformations, crimping or similar deformations all along the thread. J. W. B.

2-Sulphoalkyl Ethers of Cellulose—Textiles having Ion-exchange Properties, Organdie Finish. U.S. Secretary of Agriculture. *USP* 2,681,846

When a cellulosic textile containing reactive hydroxyl groups is treated with mercerising lye containing > 15% of a sulphoalkylating agent, cured under moderate conditions and washed free of alkali, it remains fibrous and insoluble in water and consists of the alkali metal salt of a sulphoalkyl cellulose ether. It has the ion exchange properties of an insoluble strongly acidic material containing monobasic acid groups. The fibres have strong cation affinity, thus they have good affinity for basic dyes and the ions of nitrogenous bases and the materials so produced have improved resistance to burning. This process imparts an organdie finish to fabrics and the fabrics are highly reactive with basic reagents, e.g. metal salts used for rotproofing. C. O. C.

Laminated Fabrics. Trubenised (Gt. Britain). *BP* 732,107

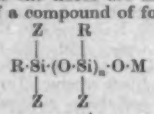
In a fabric composed of both thermoplastic and non-thermoplastic yarns which is used, e.g. as the interlayer for bonding two other fabrics together, use as the thermoplastic fibres of a material which is solid at atmospheric temperature but becomes adhesive when heated, e.g. polythene, enables bonding to be accomplished by heat and pressure alone. C. O. C.

Laminated Semi-stiff Fabrics. Ward, Brooke & Co. *BP* 732,979

An interlining containing a cellulose derivative is placed between two fabrics and the assembly immersed in industrial methylated spirit containing a little ethyl acetate. When the cellulose derivative is softened the assembly is taken out of the liquor and hot pressed. The laminated fabric produced is treated with an aqueous solution of a fluorescent brightening agent in presence of Na_2SO_4, rinsed and dried. The interlining before being placed between the fabrics to be bonded may be sprayed on one or both sides with a dispersion of ultramarine. The resulting fabric retains its colour longer than similar known fabrics. C. O. C.

Improving the Adhesion of Resins to Glass Fibres. Owens-Corning Fiberglas Corpn. *USP* 2,683,097

Improved adhesion between resinous coatings and glass fibres is obtained if the fibres are first treated with an aqueous solution of a compound of formula—



(M = alkali metal or ammonium; one or preferably all of the R groups = unsat. radical having < 6 C in a straight chain; Z = OM or R), e.g. Na allylpolysiloxanolate. C. O. C.

Cotton Slashing with Synthetic Compounds as a means towards Pollution Abatement (II p. 551).
Animalising Cellulose with *iso*Melamine Resins (VI p. 560).
Colouring and Wet Processing of Fabrics containing Fibrolane Regenerated Protein Fibre (VIII p. 562).
Combined Embossing and Printing (IX p. 563).
Permanent Local Mechanical Effects on Crêpe Fabrics (IX p. 563).
Diazotype Printing Materials (IX p. 563).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Permanence of Paper—IV. W. H. Langwell. *Proc. Tech. Sectn. British Paper & Board Makers' Assocn.*, **36**, 199–207 (Feb. 1955).

A wide range of British commercial writing and printing papers were tested for liability to damage by atmospheric sulphur dioxide, and a suggested standard accelerated ageing test was devised. Dilute ethylenediaminetetra-acetic acid was found to be a practical inhibitor applicable to the whole range of samples investigated. S. V. S.

Structure of Native and Mercerised Celluloses. C. Legrand, A. Bonnemayre, A. Guinier, and P. Antzenberger. *Nature*, **175**, 1045 (11 June 1955).

X-Ray studies of celluloses by two complementary methods did not give the interference result obtained by Sen and Roy. Irradiation of fibres with a beam from a tube with a chromium anode gave the spots noted by Sen and Roy, but these disappeared on monochromatisation of the beam. W. R. M.

Mercerisation of Cellulose. IV—Phase Transition Studies on Technical Wood Pulps and Cotton Linters. B. G. Rånby and H. F. Mark. *Svensk Papperstidning*, **58**, 374–382 (31 May 1955).

The mercerisation of 10 different hardwood sulphite and sulphate pulps and one chemical-grade cotton linters pulp was studied as a phase transition process using water sorption measurements and X-ray diffraction analysis. The water sorption transition curves were measured on both the adsorption and the desorption isotherms, and showed the same transition interval and the same slope without hysteresis effect. The slope is highest for the linters pulp and lowest for the sulphate pulps. Sulphate pulps have higher resistance to swelling with caustic soda than sulphite. Prehydrolysis of the wood chips appeared to give increased resistance to swelling of the sulphate pulps. S. V. S.

Hemicellulose extracted from Cellulose by Water. G. W. Huffman, P. A. Rebers, D. R. Spriestersbach, and F. Smith. *Nature*, **175**, 991 (4 June 1955).

Extraction of filter paper, cotton linters, and wood α-cellulose by water gives a soluble carbohydrate, which appears to be a pentosan containing uronic acid units as well as neutral sugars. This may interfere with micro-quantitative paper chromatography and in the separation of substances by sheet paper and cellulose column chromatography. W. R. M.

Nature of Adsorption on Cellulose from Aqueous Solutions. C. H. Giles, S. K. Jain, and A. S. A. Hassan. *Chem. and Ind.*, 629–630 (28 May 1955).

The adsorption properties of cellulose and chitin towards various solutes in solution are examined. It is concluded that—(1) Simple hydroxy compounds are adsorbed by both substrates by hydrogen bonding in the absence of water. (2) The same compounds are adsorbed by chitin from aqueous solution by hydrogen bonding, but are unadsorbed by cellulose because of preferential adsorption of water. (3) Simple sulphonic acids are not adsorbed by chitin if salt formation is prevented by a buffer, but some are adsorbed by cellulose. (4) Free sulphonic acids, without hydrogen-bonding substituents, are adsorbed by van der Waals forces. (5) Compounds of similar molecular area but differing numbers of hydrogen-bonding groups are adsorbed to a similar extent by cellulose irrespective of the substituent groups, but on chitin adsorption varies. (6) The planar structure of cellulose permits the close approach of solute necessary for van der Waals adsorption, but the less planar structure of chitin does not. Finally, it is concluded that cellulose adsorbs solutes from water by van der Waals attraction and not by hydrogen bonding. W. R. M.

Reduction of Aldehyde Groups in Periodate Oxycelluloses by Sodium Borohydride. F. S. H. Head. *J. Textile Inst.*, **46**, T 400–T 406 (June 1955).

When periodate oxycelluloses are treated with aqueous sodium borohydride their reducing power, as indicated by the copper number, and their fluidity in cuprammonium solution are greatly diminished. It appears that the aldehyde groups are reduced by the borohydride, and that the reduction leads to stabilisation of linkages which the aldehyde groups made alkali-sensitive. The greatest effect is shown with unbuffered solutions (final pH up to about 10·5) with a borohydride concentration of not < 0·01 M; this is about 16 times the theoretical requirement when using 50 ml. per gram of oxycellulose. A treatment of 24 hr. at 20°C. reduces the fluidity from 41·8 to 6·3; higher temperatures are less effective. J. W. B.

Oxidation of Aldehyde Groups in Periodate Oxycelluloses by Chlorous Acid. G. F. Davidson and T. P. Nevell. *J. Textile Inst.*, **46**, T 407–T 423 (June 1955).

Periodate oxycelluloses are treated with acidified chlorite solutions; first of all, data are presented on the effect of pH and chlorite concentration on the decomposition of chlorite solutions, and show that the half-life of a 0·1 M solution at pH 1·3 and 3 is 0·5 and 72 hr. respectively, and at pH 3 the half-life for 0·1 M and 0·2 M. solutions is 72 and 29 hr. respectively. The reaction with aldehyde is most complete at pH 3, though the initial rate is slower than at lower pH values, at which, however, the reagent decomposes before the reaction is complete. Also, at the lower values appreciable hydrolytic scission of the chain molecules takes place. Results also show that the active agent is undissociated chlorous acid and not chlorine dioxide, as a decomposed solution which contains chlorine dioxide only has no effect. The optimum temperature, to avoid chain scission, is 20°C., but the optimum chlorite concentration depends on the extent of the periodate oxidation; in any case, a considerable excess over the theoretical amount is required. The reagent has very little effect on unoxidised cellulose. The chlorite treatment is then applied to 16 periodate oxycelluloses, and it is found that the final products contain an average of 1·78 COOH groups for every O atom consumed in the periodate oxidation. This is a higher yield than previously obtained but lower than the theoretical value of 2, and reasons are tentatively advanced to explain this. The relation between copper number and aldehyde content, as measured by the difference in COOH content before and after maximum conversion by chlorite, is studied, and evidence is presented suggesting that one of the two aldehyde groups in a chain unit of a periodate oxycellulose is preferentially oxidised by chlorous acid and that this group reduces more copper in the copper number determination, and contributes more to the alkali sensitivity of the oxidised chain molecule, than the other.

J. W. B.

Chemical Reactions in the Making of Cellulose Acetate. C. J. Malm and L. J. Tanghe. *Ind. Eng. Chem.*, **47**, 995–999 (May 1955).

The basic reactions of acetylation, deacetylation, sulphation, and desulphation, which occur in the manufacture of cellulose acetate from cellulose, acetic anhydride, and sulphuric acid, have been studied individually. In the esterification step, there is competition between the acetylation and sulphation reactions for the cellulose hydroxyl groups: both have preference for the primary hydroxyls. Sulphation is very rapid compared with acetylation, but at the end of esterification slow desulphation occurs, sulphate being replaced by acetyl. In the hydrolysis stage, the optimum amount of water for removal of sulphate is $< 1\%$ of the acetic acid, but deacetylation requires much more water. Both reactions are reversible. Hydrolysis is the net result of deacetylation and reacetylation. With increasing concentration of water, reacetylation is retarded, and more primary hydroxyl is found in the product.

W. K. R.

Use of Activated Cellulose for the Preparation of Methyl- and Ethyl-celluloses of Low Alkyl Content. E. N. Lyubimova and N. I. Nikitin. *J. Appl. Chem. U.S.S.R.*, **28**, 402–406 (April 1955).

By the methylation and ethylation of mercerised wood cellulose activated by freezing in 8% NaOH and by mild xanthation (introduction of one CS·SNa per 40 glucose residues), products are obtained varying in D.P. and in alkoxyl content (1–18% $O{\cdot}CH_3$ and 0·8–7·7% $O{\cdot}C_2H_5$). For a given D.P. and alkoxyl content, methyl- and ethyl-celluloses prepared in this way are much more sol. (in water and in 6% NaOH respectively) than products obtained from unactivated cellulose.

A. E. S.

Degradation of Carbohydrates by Alkali. IX—Cellobiose, Cellobiulose, Cellotetraose, and Laminarin. W. M. Corbett and J. Kenner. *J.C.S.*, 1431–1435 (May 1955).

Cellobiose and cellobiulose are degraded by alkali similarly to degradations described in *J.C.S.*, 2245 (1953) and 1789 (1954), whereas cellotetraose and laminarin undergo peeling processes typical of the course of alkaline degradation of hydro- and oxy-celluloses.

H. H. H.

PATENTS

Mineral-coating Composition for Paper. S. D. Warren Co. *USP* 2,685,538

Using an aqueous dispersion of latex produced by the emulsion polymerisation of an ethylenically unsaturated monomer, e.g. copolymer of 60% of styrene and 40% *iso*prene, yields a composition which gives a coating of improved resistance to water. The composition can be used at a high solids content. The resultant coating is superior to coatings containing casein as the adhesive in smoothness, flatness, calendered and uncalendered gloss, flexibility, fold, printing, ink setting, curl and velvety feel.

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C. O. C.

Transfer Material. National Cash Register Co. *BP* 732,284

Incorporation into the transfer film of 0·5–10·0% of the volume of the film of hard particles of average diameter 1–3 times the thickness of the film prevents marking off when sheets of paper coated with easily rupturable material are stacked under pressure.

C. O. C.

Absorbent Cellulosic Products (X p. 564).

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Resistance of White Leather to Breakdown by Perspiration. W. T. Roddy and R. M. Lollar. *J. Amer. Leather Chem. Assocn.*, **50**, 180–192 (April 1955).

The hydroxy acid components of perspiration cause detannage of chrome leather, leaving the fibres unprotected. Actual deterioration of the leather fibres is associated with the presence of lactic acid in the perspiration. Damage occurs more readily with white leather shoes than with black or brown, as the latter receive longer protection by virtue of a higher Cr content. Mould growth or other bacterial action has no effect on the stability of the tannage under these conditions.

J. W. D.

Mechanism of Unhairing Skins and Hides by means of Certain Proteolytic or Amylolytic Enzymes. S. M. Bose, W. M. Krishna, and B. M. Das. *J. Amer. Leather Chem. Assocn.*, **50**, 192–199 (April 1955).

The hydrolysis of both fibrous and non-fibrous skin proteins by protease (from madar latex), diastase, and the amylase of "ragi" has been studied under controlled conditions. All the non-fibrous proteins are readily hydrolysed by the protease, but some of the fibrous ones, e.g. collagen and keratin, are unaffected; other fibrous proteins such as reticulin and elastin are only slightly hydrolysed. The mucoids, albumin, and globulin are readily hydrolysed by amylolytic enzymes also, yielding reducing sugars. Enzymic unhairing of these types thus consists in the hydrolysis and removal of mucoid material.

J. W. D.

Decomposition of Cystine in Acid Solution in the Presence of Indoles. J. T. Edward and E. F. Martlew. *J.C.S.*, 1636–1637 (May 1955).

The formation of cysteine from cystine in 7 N-HCl is catalysed not only by tryptophan—cf. Olcott and Fraenkel-Conrat (*J. Biol. Chem.*, **171**, 583 (1947))—but by other indoles (e.g. skatole and 2-methylindole) and by xanthhydrol, although not by diphenylamine. Paper-chromatographic examination of the reaction products revealed the presence of several other ninhydrin-positive compounds, of which two appeared to be serine and alanine. The decompositions seem to be acid–base-catalysed reactions.

H. H. H.

PATENT

Leather Finish containing Caprolactam and a Resinous Polymer. DuP. *USP* 2,686,764

An aqueous emulsion containing caprolactam and a macromolecular polymer of a monomeric olefinically unsaturated compound, e.g. butyl methacrylate resin, spreads and penetrates into leather in a very efficient manner. C. O. C.

Nature of Adsorption on Cellulose from Aqueous Solutions (XI p. 565).

XIII—RUBBER; RESINS; PLASTICS

Hysteresis Phenomena in Unimolecular Polyamide Films. H. Schuller. *Kolloid-Z.*, **136**, 134–136 (May 1954).

The first compression of a fresh film of poly-ε-caprolactam on water gave a force–area curve which lay above the hysteresis loop obtained on subsequent expansions and compressions. Increase of temperature raised the loop to higher pressures. Irradiation with ultraviolet radiation increased the pressure but eliminated hysteresis. This effect was reversible. Apparently the irradiation broke the intermolecular bonds responsible for hysteresis. W. R. M.

By-products of the Beckmann Rearrangement of *cyclo*Hexanoneoxime—I. A. Schäffler and W. Ziegenbein. *Chem. Ber.*, **88**, 767–772 (June 1955).

From crude ε-caprolactam roughly 0·1% *cyclo*hexanone, 0·001% aniline, and 0·0001% octahydrophenazine are isolated, and phenyl *iso*cyanide is shown to be present. H. E. N.

Heat and Light Degradation of Vinyl Chloride Resin. L. H. Wartman. *Ind. Eng. Chem.*, **47**, 1013–1019 (May 1955).

The discoloration which occurs on exposure of vinyl chloride resin compositions to heat or ultraviolet radiation is minimised by the presence of stabilisers, which are compounds capable of reacting with the HCl formed by degradation of the resin. An analytical method is described for measuring the amount of chloride reaction products, and hence the consumption of stabiliser, and the results are correlated with the extent of discoloration on heating and light exposure. The efficiency of various stabilisers as HCl acceptors has also been determined by measuring the amount of HCl which escapes when a stabilised resin film is heated. The discoloration of vinyl chloride resin in the presence of stabilisers is a complicated and little understood phenomenon; slow HCl pick-up by a stabiliser is only one cause of poor colour retention. W. K. R.

Detection of Free Radicals in Polyacrylonitrile by Paramagnetic Resonance. C. H. Bamford, A. D. Jenkins, D. J. E. Ingram, and M. C. R. Symons. *Nature*, **175**, 894–895 (21 May 1955).

Trapped free radicals in the polymerisation of acrylonitrile are detected and estimated by observation of the paramagnetic resonance absorption spectrum of the polymer. Only 1–2% of the total number of radicals generated become trapped. W. R. M.

Synthesis of Benzyl Ethers of Polyvinyl Alcohol. S. N. Ushakov and E. M. Lavrent'eva. *J. Appl. Chem. U.S.S.R.*, **28**, 407–413 (April 1955).

Polyvinyl alcohol is benzylated with benzyl chloride in presence of NaOH, and products having degrees of substitution (DS) of 10–80% are obtained (complete substitution = 100%). The DS attained increases regularly as the concn. of NaOH increases from 5 to 40%. With increase in DS the vitrification temp. of the polymer falls from 85°c. (DS = 0) to 13°c. (DS = 80%). Films of the products are examined for their mechanical properties and permeability to water vapour. A. E. S.

PATENTS

Antioxidants for Rubber. ICI. *BP* 732,928

αβ-Bis-(2-hydroxy-5-methylphenyl)-ethanes containing in the 3-position of each of the two phenyl radicals an alkyl, *cyclo*alkyl or alkyl*cyclo*alkyl group of < 9 C, e.g. 1:2-bis-(2-hydroxy-3:5-dimethylphenyl)-ethane, are excellent antioxidants for natural and synthetic rubbers. C. O. C.

Decorative Sheet Rubber. Dunlop Rubber Co. *BP* 732,839

Particles of cured crumb rubber are pressed into the surface of a backing sheet of uncured rubber. The sheet is then vulcanised in a vacuum mould with the external surface of the crumbs lying in the same plane as the surface of the backing sheet. C. O. C.

Readily Dyed Polyacrylonitrile (VI p. 561).

XIV—ANALYSIS; TESTING; APPARATUS

Review of Industrial Applications of Analysis, Control, and Instrumentation. *Anal. Chem.*, **27**, 574–690 (Part II, April 1955).

Copiously referenced reviews under 14 headings; these include— natural and synthetic rubbers, coatings, pesticides, and water analysis. J. W. D.

Qualitative and Quantitative Analysis of Mixtures of Surface-active Agents with special reference to Synthetic Detergents. C. Kortland and H. F. Dammers. *J. Amer. Oil Chem. Soc.*, **32**, 58–64 (Feb. 1955).

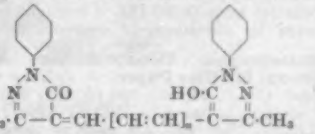
An identification scheme for surface-active agents is given which enables these agents or their mixtures to be detected in commercial products. Use is made of the acid resistance of the various types to separate the different compounds step by step from the mixture. An important feature of the method is that it can be used also for quantitative analyses by weighing the extraction residues obtained and determining their chemical and physical constants. P. G. M.

Complexes of Eriochrome Black T with Calcium and Magnesium. A. Young and T. R. Sweet. *Anal. Chem.*, **27**, 418–420 (March 1955).

Studies based on the continuous variation of metal content at each of a number of different pH values indicate that 1 : 1, 2 : 1, and 3 : 1 complexes exist for both Ca and Mg. This is a wider range than has previously been reported. It is suggested that the 3 : 1 complexes may result from the functioning of the dye anion as a bidentate through the two phenolic oxygens, or through one azo nitrogen and an adjacent phenolic oxygen; the metal ion would thus have a co-ordination number of 6, forming an octahedral complex in which the sp^3d^2 orbitals of the metal are involved in bond formation. J. W. D.

New Organic Reagent for Silver and Copper. B. Gehauf and J. Goldenson. *Anal. Chem.*, **27**, 420–421 (March 1955).

A red dye ("Zolon Red") prepared from 1-phenyl-3-methylpyrazolone, pyridine, NaCN, and Chloramine-T gives deep blue cpd. with Ag^+ and Cu^+ ions. The sensitivity is 1 part in 600,000 for Ag, and 1 part in 250,000 for Cu. It is suggested that the dye has a polymethin-type structure, closely related to—



J. W. D.

Spot Reaction for Acidic Polynitro Compounds. F. Feigl and V. Gentil. *Anal. Chem.*, **27**, 432–433 (March 1955).

Enolisable polynitro compounds yield red–pink salts, whose soln. in benzene fluoresce orange in ultraviolet radiation, when their weakly alkaline soln. are shaken with an acid soln. of Rhodamine B and benzene–ether. The identification limits of 8 cpd. are given. The presence of nitro and carboxyl groups in enolisable nitro cpd. improves the sensitivity of the test, by increasing the stability of the red salt soln. J. W. D.

Phenol-indo-2:6-dichlorophenol as a Spray Reagent. J. Barnabas and G. V. Joshi. *Anal. Chem.*, **27**, 443–444 (March 1955).

The dye, phenol-indo-2:6-dichlorophenol, gives good differentiation amongst certain organic acids, when used as a spray reagent with their chromatograms. J. W. D.

New Adsorption Analysis for Carotenes in the Pigments of Green Leaves. R. Köppen. *Kolloid-Z.*, **135**, 150–159 (March 1954).

Previous methods for carotene estimation in dry green fodders are comprehensively discussed with respect to the difficulties involved. The latter are then overcome by a new and rapid method which is based on shaking a solution of the leaf green pigments in benzine, light petroleum, or pentane with $CaCO_3$ which has been pptd. from aq. $CaCl_2$ by aq. NaOH in slight excess and containing sufficient water-glass to give 10% SiO_2 in the ppt.; the latter is dried first on the water-bath and then at 150°c. All the pigments with the exception of carotene are adsorbed by the active $CaCO_3$, and, after centrifuging the mixture, the carotene is directly determined in the filtrate. 11 references. H. H. H.

Bromine Allwörden Reaction. R. D. B. Fraser and G. E. Rogers. *Biochim. Biophys. Acta*, **16**, 307–316 (March 1955).

The nature of the membranes defining the bubbles in the bromine Allwörden reaction has been studied using the gold shadowing technique in optical microscopy. In merino wool the membrane is shown to consist of the epicuticle with an adhering component of the exocuticle. The unsymmetrical development of bubbles in merino wool has been reinvestigated and shown to be a transient phase. A possible mechanism of the bromine Allwörden reaction which explains this phenomenon without assuming a bilateral cuticle structure is presented. The variable reaction obtained with coarse wools and hair and the course of the reaction in merino wool are discussed in terms of this mechanism. P. G. M.

Differential Measurements of Reflectance. C. A. Lermond and L. B. Rogers. *Anal. Chem.*, **27**, 340–346 (March 1955).

Reflectance measurements in which a standard spectrophotometer was used, together with a photomultiplier, show that small differences can be accurately measured in regions of low reflectance. Numerous results are cited, shown as graphs, and discussed, illustrating the application of the method to solid mixtures (including pigments), yarn dyeings, and prints and dyeings on cloth. The strength and closeness of match of dyeings can be accurately assessed, and variations have been shown in the evenness of printed stripes that are barely detectable visually. It is possible to differentiate readily between lowering of reflectance resulting from the presence of neutral greyness and that resulting from an increase in colorant concn. Various methods of plotting the results are discussed. J. W. D.

Colour Matching in Artificial Light. G. Smiatek. *Textil-Praxis*, **10**, 387–391 (April 1955).

Discussion of theoretical aspects of matching in lights of various sources, together with a description of the xenon high-pressure lamp XBO 162, a daylight matching lamp unaffected by variations in current. B. K.

Chromatographic Determination of Carboxyl Groups in Filter Paper. A. J. Ultee and J. Hartel. *Anal. Chem.*, **27**, 557–560 (April 1955).

The method is based on that of Schute (*Nature*, **171**, 839 (1953)); Pb^{2+} ions are used to associate with the COOH groups, these giving the most satisfactory results amongst the metals investigated. High concn. of lactone groups cause some difficulty. The mechanism of the reactions involved has been examined by infrared spectroscopy. J. W. D.

Determination of Acetovanillone in Oxidised Alkaline Cleaved Sulphite Liquor by Paper Chromatography. E. T. Reaville and G. W. Shreve. *Anal. Chem.*, **27**, 565–566 (April 1955).

The chromatograph is developed with *n*-butyl ether, and a band of $NaHSO_3$ is used to block the movement of vanillin and *p*-hydroxybenzaldehyde, but not that of acetovanillone or vanillil. The acetovanillone band is located, after brief exposure to ammonia, by ultraviolet radiation, is cut out, and is leached with a phosphate buffer at pH 7·3. Absorbance measurements at five specified wavelengths permit determination of the acetovanillone present, and correction can be made for any vanillil. J. W. D.

Determination of Combined Acetic Acid Content of Cellulose Acetate—Gravimetric Method. G. Garetto and A. Ruffoni. *Anal. Chem.*, **27**, 400–401 (March 1955).

The combined acetic acid content of acetone-soluble cellulose acetate may be determined by weighing the cellulose regenerated from the ester by means of complete saponification in an aqueous–alkaline medium. The accuracy of the method compares favourably with that of the usual volumetric methods. J. W. D.

Volumetric Determination of Cellulose Nitrate and Nitroguanidine by Transnitration of Salicylic Acid. H. Stalcup and R. W. Williams. *Anal. Chem.*, **27**, 543–546 (April 1955).

The nitrogen content of cellulose nitrate may be determined accurately (standard deviation $\pm$ 0·02%) by using the material to effect the transnitration of salicylic acid in H_2SO_4 soln., the resulting nitrosalicylic acid being subsequently reduced with standard $TiCl_3$. J. W. D.

Determination of Carboxylic Amides by Reduction to the Corresponding Amine. S. Siggia and C. R. Stahl. *Anal. Chem.*, **27**, 550–556 (April 1955).

Primary, secondary, and tertiary amides may be determined by reduction to the corresponding amines, using $LiAlH_4$; the amine formed is steam-distilled from the reaction mixture and titrated with standard acid. The method, the precision of which is within $\pm$2% (or better), is applicable to amides ranging from those of low mol. wt. to those of the fatty acids. Two methods are described, one using a visual end-point, the other a potentiometric one. J. W. D.

Limits of Error in the Viscosimetry of Solutions of Macromolecular Substances. H. Frind. *Faserforsch. und Textiltech.*, **6**, 25–28 (Jan. 1955).

Errors arising from visual timing are such that for the error in the intrinsic viscosity to be within $\pm$ 1–2% the relative viscosity must be $\leq$ 1·05 when an Ubbelohde viscometer is used and $\leq$ 1·02 when an Umstätter viscometer is used. A. E. S.

Accuracy of Viscosity Measurements on Very Dilute Solutions. F. Winkler. *Faserforsch. und Textiltech.*, **6**, 29–32 (Jan. 1955).

Analysis of the possible errors in the measurement of the sp. viscosity of a dilute soln. shows that such determinations can be very inaccurate. Little advantage can be gained by increasing the accuracy of timing, but some improvement can be effected by carrying out numerous parallel determinations. A. E. S.

Measuring the Detergent Effect of Soaps, etc. American Conditioning House. *USP* 2,684,595

Apparatus which reproduces on small pieces of fabric the effect of commercial laundering machines. C. O. C.

Electronic Analysis of Scientific Data. T.M.M. (Research). *BP* 732,790

Apparatus for use in the analysis of data recorded in the form of traces on charts, comprises a cathode ray tube in which a light spot travels across the screen; its image is projected on the chart so that the spot is obscured when it reaches the trace. This causes a response in a photo-electric cell, and the time taken between the spot leaving a datum line and reaching the trace is a measure of the length of the ordinate. Means are provided for giving an electric output proportional to the ordinate; this output may be applied to any suitable electronic analysing or integrating instrument. J. W. B.

Colour Reactions of Unsaturated Carbonyl Compounds. II—Use of *p*-Aminodimethylaniline (IV p. 553).
Crystalline–Amorphous Ratio of Keratin Fibres. I—Hailwood–Horrobin Analysis of the Water-adsorption Isotherms (VI p. 539).
Crystalline–Amorphous Ratio of Keratin Fibres. II—Hydrogen–Deuterium Exchange Reaction (VI p. 559).
Permanence of Paper—IV (XI p. 565).
Hemicellulose extracted from Cellulose by Water (XI p. 565).
Oxidation of Aldehyde Groups in Periodate Oxycelluloses by Chlorous Acid (XI p. 566).

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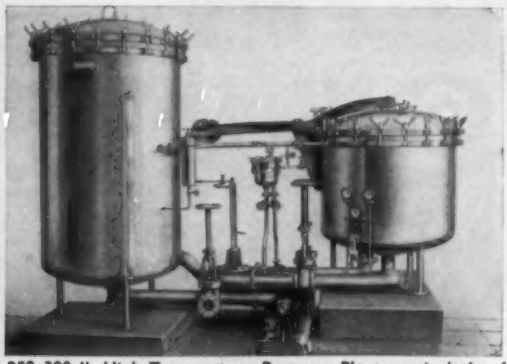

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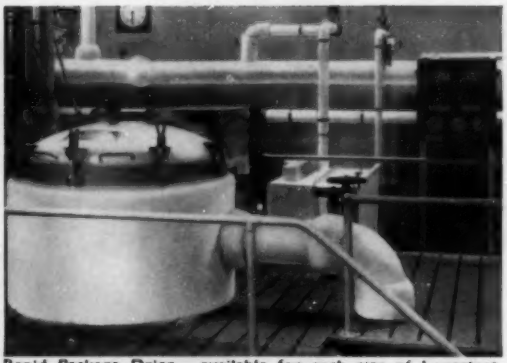

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Castle, W. C., formerly c/o Clayton Dyestuffs Co. Ltd., Manchester, to c/o Ciba Co. Pty. Ltd., P.O. Box 22, East Brunswick N.11, Victoria, Australia

Cryer, N., formerly of 1294 Boundary Lane, Hatfield, Pretoria, S. Africa, to 21 Lawley Street, Waterkloof, Pretoria, South Africa

Drzewidki, M., formerly of 105 Westminster Road, Morecambe, to 3 Ruskin Drive, Bare, Morecambe

Evans, D. C., formerly of Messrs. Wm. Tatton & Co. Ltd., Upper Hulme, near Leek, Staffs., to Brindle Coombe, Westwood Park Drive, Leek, Staffs.

Faulds, W., formerly of 128 Hollingwood Lane, Bradford 7, to 12 Back Lane, Holmfirth, near Huddersfield

Gee, N., formerly of 92 Turves Road, Cheadle Hulme, Cheshire, to 6 Evington Street, Leicester

Gibson, F., formerly of 68 The Hollins, Sowerby Bridge, Yorks., to 43a Wharf Street, Sowerby Bridge, Yorks.

Goodwin, J. E., formerly of Wycliffe House, Watbourne Park, Urmston, to Rathcar, 9 Houghton Lane, Swinton, Lancashire

Hannay, G. J., formerly of Hindshaw, Lester Dyestuffs Ltd., 38–46 Sunbridge Road, Bradford, to Industrial Dyestuffs Ltd., Cater Buildings, Cater Street, Bradford

Heywood, D. W. (2752198), formerly of 33 Heron Street, Pendlebury, Manchester, to Hut 376/E, F.C.I. 35, 3 Wing, No. 3 Radio School, Compton Bassett, Calne, Wilts.

Hindshaw, N., formerly of 27 Brownside Road, Cambuslang, to 9 Kings Park, Knock, Belfast, N. Ireland

Houghton, F. R., formerly of Wingfield, Ash Grove, Otley, to Moorview, 23 Tranmere Drive, Guiseley, near Leeds

Kerr, C. D., formerly of 19 Knockbracken Park, Ormeau Road, Belfast, to 6 Daleview Drive, Clarkston, Renfrewshire

Kerr, N., formerly of "Smithfield", Glasgow Road, Dumbarton, Scotland, to c/o 47 The Square, Droylsden, Manchester

Kitchin, J. B., formerly of I.C.I. Ltd., Dyestuffs Div., Trafford Park Works, Westinghouse Road, Trafford Park, Manchester, to c/o Imperial Chemical Industries Ltd., Centralwork Study Dept., Imperial Chemical House, Millbank, London S.W.1

Lascaris, L., formerly of 8 Queens Road, Edgerton, to 23 Thorp Avenue, Fieldhouse, Holmfirth, Huddersfield

Lewis, G., formerly of 3 Cross Brook, Todmorden, Lancs., to 41 Laund Hill, Belper, Derbyshire

Lockhead, G. T., formerly of 157 Nansen Road, Leicester, to 23 Ashby Tree Road, Oadby, Leicestershire

Manitius, L. G., formerly of 50–57th Avenue, Pointe Aux Trembles, Montreal 5, Quebec, Canada, to 8606 de Grosbein Street, Montreal 5, Quebec, Canada

Mills, B. W., formerly of 1 Daynier Street, Leek, Staffs., to 1 Dampier Street, Leek, Staffs.

Mooradian, S., formerly of American Cyanamid Co. Ltd., Bound Brook, New Jersey, U.S.A., to 225 North Bridge Street, Somerville, New Jersey, U.S.A.

Oakes, T., formerly of 24 Neptune Boulevard, Strathmore, Quebec, to c/o North American Cyanamid Co. Ltd., 5550 Royalmont Avenue, Town of Mount Royal, Quebec, Canada

Palmer, L. V. C., formerly of 69 Hatherley Gardens, East Ham, London E.6, to 19 Llanerch Crescent, Gorseinon, near Swansea, Glamorgan

Pickin, R., formerly of 94 Quarry Avenue, Bebington, Wirral, Cheshire, to 30 Francis Street, Heckmondwike, Yorks.

Reis, A. A. C., formerly of 10 Blenheim Road, Manningham Lane, Bradford, to Travessa dos Namorados, No. 1, Covlha, Portugal

Rolfe, E., formerly of Fern Leigh, Far End Lane, Honley, Huddersfield, to Thorney Mell, 28 Chester Road, Poynton

Ruppenthal, K. C., formerly of Pittsburgh Coke & Chem. Co. Ltd., Fine Chem. Div., Pittsburgh 25, Pennsylvania, U.S.A., to Pittsburgh Coke & Chemical Co. Ltd., Fine Chemicals Division, P.O. Box 408, Spartanburg, South Carolina, U.S.A.

Sadowski, J. Z., formerly of 28 King Street, Dukinfield, Manchester, to 24 Claremont Road, Manchester 16

Sheard, G. E., formerly of 15 West Royd, Rodley Lane, Bramley, Leeds, to 4 Southlands Road, Riddlesden, Keighley, Yorks.

Stephenson, Mrs M., formerly of 46 Mountside Crescent, Prestwich, Manchester, to 50 Park Avenue, Sale, Manchester

Strudwick, J. W., formerly of 7 Woodhall Park Drive, Stanningley, near Leeds, to "Manderville", Wynmore Avenue, Bramhope, near Leeds

Taylor, T. P., formerly of 40 Manchester Road, Chorlton-cum-Hardy, Manchester, to 534 Welbraham Road, Chorlton-cum-Hardy, Manchester 21

Terrington, D., formerly of 23 Fern Street, Boothtown, Halifax, to Shree Digvijaya Woollen Mills Ltd., Aerodrome Road, Jamnagar, India

Thomas, W. L., formerly of Hill Crest, Carlton Drive, Heaton, Bradford 9, to Hill Crest, Leylands Lane, Heaton, Bradford 9

Wilkinson, C., formerly of 77 Baildon Road, Baildon, Yorks., to Messrs. Wilkinson & Scott Ltd., 58 Nelson Street, Bradford 5

ADDRESSES WANTED

Alcada, J. C., formerly of 9 Mornington Villas, Flat No. 4, Bradford 8

Crook, B., formerly of 196 Wentworth Street, W., Magog, Quebec

Crowther, D., formerly of 551 Leeds Road, Thackley, Bradford

Dickinson, K., formerly of 16 Hollycroft, Hinckley, Leics.

Ferguson, T. D., formerly of P.O. Box 106, Altavista, Virginia, Mass.

Green, A. N., formerly of 19 Salcombe Road, Basford, Nottingham

Kesprek, A. E., formerly of 15 Huntley Street, Hamilton Street, Ontario, Canada

Meier, P. W., formerly of Oberwilerstrasse 38, Basle, Switzerland

Moore, G., formerly of 20 Welwyn Avenue, Wrose, Shipley

Morrison, A., formerly of America Woollen Co., P.O. Box 930, Lawrence, Mass., U.S.A.

Nixon, I. G., formerly of Adriaan Goekooplaan, 105 Flat Gebouw Catsheuvel, The Hague, Holland

Standring, Dr. P. T., formerly of I.C.I. Ltd., 1200 St. Alexander Street, Montreal, Canada

Stiassny, K., formerly of 70 Bialik Street, Ramat Gan, Israel

Swift, F. S., formerly of 3 St. Annes Street, Ennis, Co. Clare, Eire

a Viscosa, C. I. S., formerly of Dvoziono Centrale degh Etabiliment (Dott. Calandra), Via Sicilia 162, Roma, Italy

Warchol, W., formerly of 14 Sparford Avenue, off Wollaton Road, Nottingham

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FORTHCOMING MEETINGS OF THE SOCIETY — *continued from page* xii

Tuesday, 20th December 1955

HUDDERSFIELD SECTION. *The Dyeing of 'Courpleta'.* W. R. Beath, Esq. and J. S. Ward Esq. (Courtaulds Ltd.). Co-operative Cafe, **Huddersfield.** 7.30 p.m.

Wednesday, 11th January 1956

NORTHERN IRELAND SECTION. *The Dyeing of Terylene Polyester Fibres.* H. R. Hadfield, Esq., M.Sc. (Tech.) (Imperial Chemical Industries Ltd.). Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

Friday, 13th January 1956

LONDON SECTION. Details later.

BRADFORD JUNIOR BRANCH. Lecture Title to be announced later. Midland Silicones Ltd. Technical College, **Bradford.** 7.15 p.m.

Tuesday, 17th January 1956

HUDDERSFIELD SECTION. Title later. Professor J. B. Speakman.

SCOTTISH SECTION. *What Dyeing does to Wool.* F. F. Elsworth, Esq., Ph.D., A.R.I.C., F.S.D.C. (Wool Industries Research Association). St. Enoch's Hotel, **Glasgow.** 7 p.m.

Wednesday, 18th January 1956

MIDLANDS SECTION. Discussion Group on *Detergency.* Questions invited. G. H. Rostron, Esq., A.R.I.C. (Soap), R. C. Tarring, Esq., B.Sc., A.R.C.S. (Synthetics), and R. S. Hartley, Esq., A.R.I.C. (Research). Kings Head Hotel, **Loughborough.** 7 p.m.

Friday, 20th January 1956

MANCHESTER SECTION. *The Finishing of 'Terylene' Polyester Fibre Staple and Filament Fabrics.* G. D. Myers, Esq., B.Sc.Tech. Textile Institute, 10 Blackfriars Street, **Manchester 10.** 6.30 p.m.

Monday, 30th January 1956

BRADFORD JUNIOR BRANCH. *A New Approach to High Temperature Dyeing.* S. N. Bradshaw, Esq. (Courtaulds Coventry Ltd.). Technical College, **Bradford.** 7.15 p.m.

Thursday, 2nd February 1956

SCOTTISH SECTION. *Scouring and Milling.* B. F. J. Moxon, Esq. (Wool Industries Research Association). The Scottish Woollen Technical College, **Galashiels.** 7 p.m.

Friday, 3rd February 1956

LONDON SECTION. *A New Approach to High Temperature Dyeing.* S. N. Bradshaw, Esq. (Courtaulds Ltd., Coventry). Royal Society Rooms, Burlington House, Piccadilly, **London, W.1.** 6 p.m.

Wednesday, 8th February 1956

NORTHERN IRELAND SECTION. *General Principles in Vat Dyeing.* J. W. Reidy, Esq. (Messrs. L. B. Holliday & Co. Ltd.). Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

Tuesday, 14th February 1956

SCOTTISH SECTION. *The Dyeing and Finishing of 'Terylene' and 'Terylene' Blends.* J. G. Graham, Esq., and Dr. P. T. Standring (I.C.I. 'Terylene' Council). St. Enoch's Hotel, **Glasgow.** 7 p.m.

Wednesday, 15th February 1956

BRADFORD JUNIOR BRANCH. *Piece Scouring.* B. F. J. Moxon, Esq. (Wool Industries Research Association). Technical College, **Bradford.** 7.15 p.m.

Friday, 17th February 1956

MANCHESTER SECTION. *A New Approach to High Temperature Dyeing.* S. N. Bradshaw, Esq., B.Sc. Textile Institute, 10 Blackfriars Street, **Manchester 10.** 6.30 p.m.

NORTHERN IRELAND SECTION. Dinner Dance. Details later, at Woodbourne House Hotel.

Monday, 20th February 1956

HUDDERSFIELD SECTION. *The Finishing of 'Terylene' and 'Terylene' Wool Blended Fabrics.* N. M. Mims, Esq., B.A., B.Sc. (Joint Lecture with the Huddersfield Textile Society). Large Hall, Technical College, **Huddersfield.** 7.30 p.m.

Thursday, 23rd February 1956

MIDLANDS SECTION. *The Finishing of 'Terylene' and 'Terylene'/Wool Blends.* I. E. Haden, Esq., B.Sc., and B. I. Brooke, Esq., B.A.(Oxon.). Gas Theatre, **Nottingham.** 7 p.m.

NORTHERN IRELAND SECTION. *Film Evening.* Thompson's Restaurant, Donegall Place, **Belfast.** (Joint Meeting with the Textile Institute.) 7.30 p.m.

Friday, 2nd March 1956

LONDON SECTION. *Basic Mechanisms of Fading of Dyed Textiles.* H. B. Mann, Esq. (Courtaulds Ltd., Bocking). Royal Society Rooms, Burlington House, Piccadilly, **London, W.1.** 6 p.m.

BRADFORD JUNIOR BRANCH. Annual Dance. Connaught Rooms, **Bradford.**

Friday, 9th March 1956

MIDLANDS SECTION. MIDLANDS SECTION DINNER. Bull's Head Hotel, **Loughborough.**

Tuesday, 13th March 1956

SCOTTISH SECTION. *The Soiling of Synthetic Fibres.* G. G. Taylor, Esq., B.Sc., A.Inst.P. (Clayton Dyestuffs Co. Ltd.). St. Enoch's Hotel, **Glasgow.** 7 p.m.

BRADFORD JUNIOR BRANCH. *Some Aspects of Continuous Vat Dyeing.* Dr. R. J. Rallings (British Cotton Industries Research Association). Technical College, **Bradford.** 7.15 p.m.

Wednesday, 14th March 1956

NORTHERN IRELAND SECTION. *Various Methods of Stripping Vat, Azoic, Direct and Sulphur Dyes.* J. E. Riordan, Esq. (College of Technology, Belfast.) *Effect of pH on the Activity of Bleaching Agents.* J. Boyd, Esq. (College of Technology, Belfast). Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

Tuesday, 20th March 1956

HUDDERSFIELD SECTION. *A New Approach to High Temperature Dyeing.* S. N. Bradshaw, Esq. (Courtaulds Ltd.). Co-operative Cafe, **Huddersfield.** 7.30 p.m.

Wednesday, 21st March 1956

MIDLANDS SECTION. *Fast Dyes on Cellulose Triacetate.* J. G. Graham, Esq., B.Sc. (Joint Meeting with British Association of Chemists). Midlands Hotel, **Derby.** 7 p.m.

Thursday, 22nd March 1956

MIDLANDS SECTION. *Dyeing Faults in the Carpet and Hosiery Industries.* G. H. Lister, Esq., B.Sc., Ph.D. (Joint Meeting with the Textile Institute). Carpet Trades Canteen, **Kidderminster.** 7 p.m.

Friday, 23rd March 1956

LONDON SECTION. *Dyeing of Narrow Fabrics—Cellulosic and New Fibres.* Dr. I. M. S. Walls (I.C.I. Ltd.). George Hotel, **Luton.** 7 p.m.

Saturday, 24th March 1956

NORTHERN IRELAND SECTION. Annual General Meeting and Dinner. Details later.

Tuesday, 10th April 1956

SCOTTISH SECTION. *Textile Invention and the Dry Cleaner.* A. J. Crockatt, Esq., M.Sc., F.R.I.C. (Eastmans Dyers and Cleaners Ltd.). St. Enoch's Hotel, **Glasgow.** 7 p.m.

Saturday, 21st April 1956

BRADFORD JUNIOR BRANCH. Annual General Meeting. Technical College, **Bradford.** 10.15 a.m.

Wednesday, 25th April 1956

HUDDERSFIELD SECTION. Visit to Messrs. Stevensons Dyers Ltd., **Ambergate,** Derbys.

MIDLANDS SECTION. *A New Approach to High Temperature Dyeing.* S. N. Bradshaw, Esq. Kings Head Hotel, **Loughborough.** Preceded by the Annual General Meeting of the Section. 6.30 p.m.

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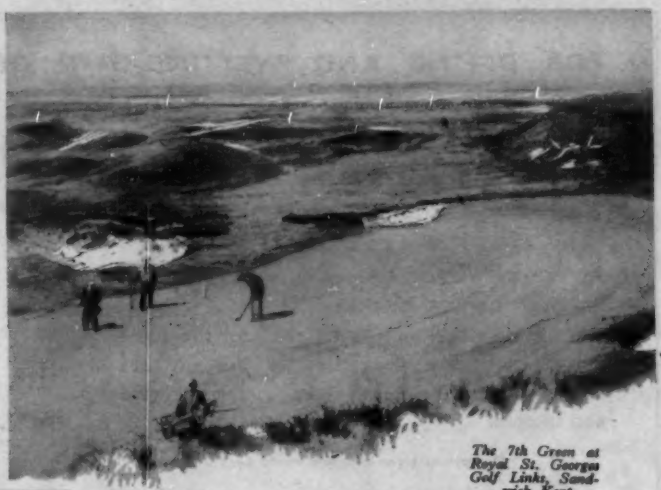

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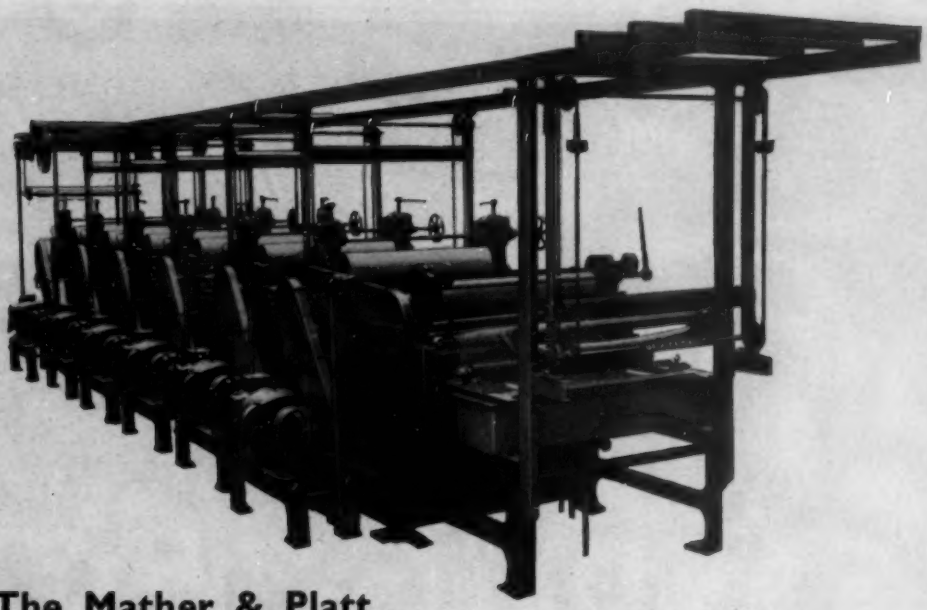

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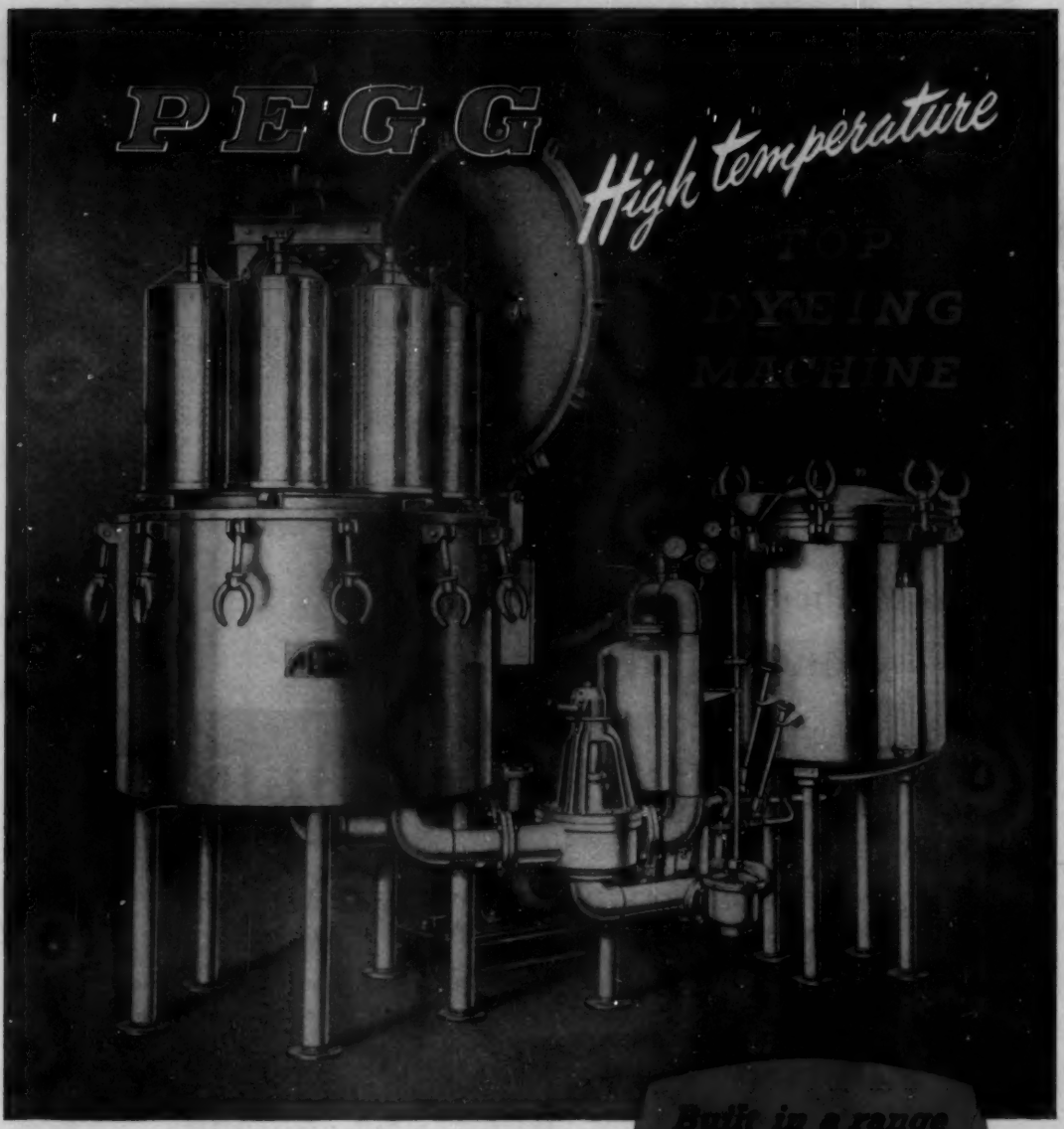

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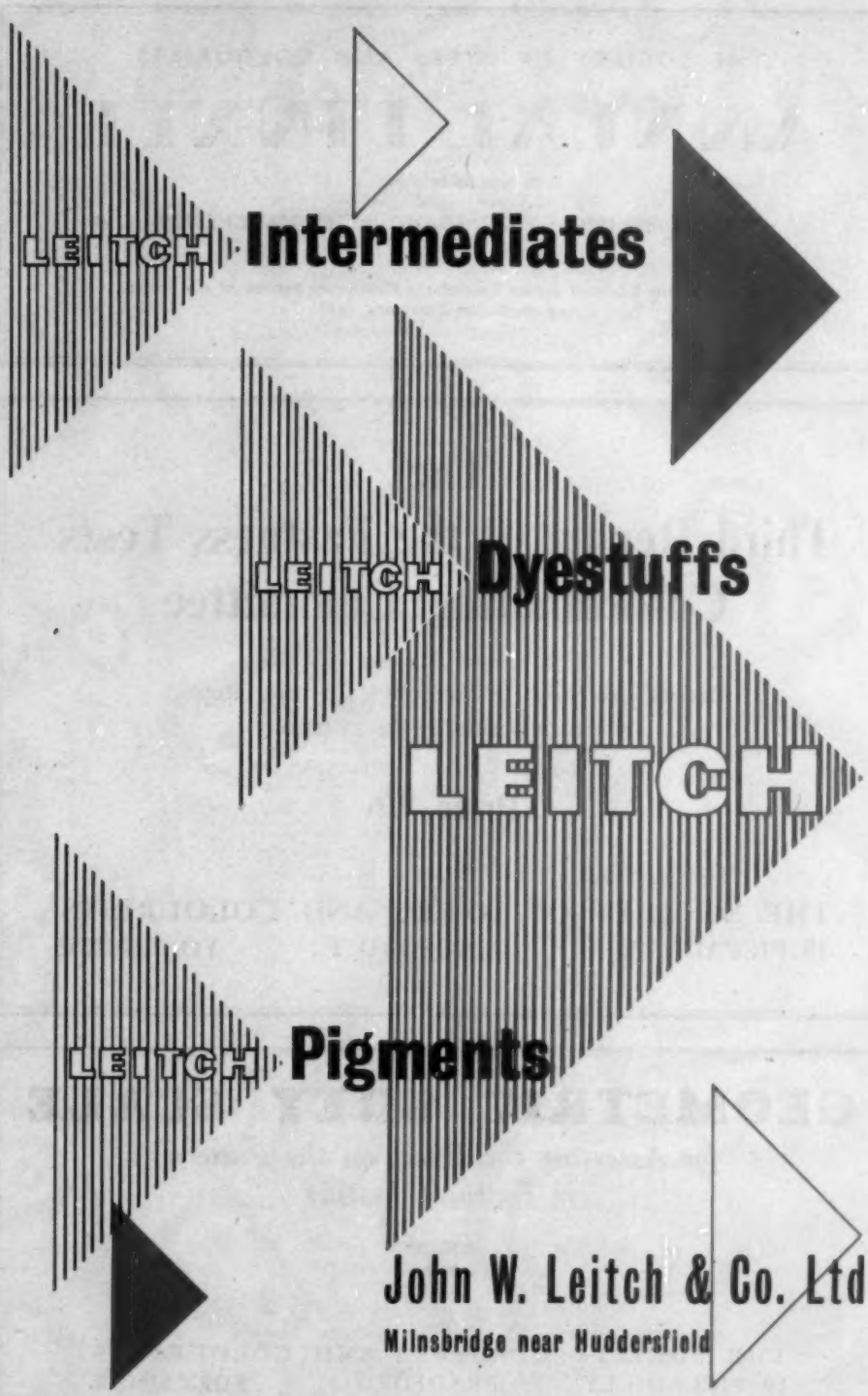
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INDEX TO ADVERTISERS

Page	
xlv	Albright & Wilson Ltd
xxxi	Alcock (Peroxide) Ltd
xl	Alginate Industries Ltd
xvii	Allied Colloids (Manufacturing) Co Ltd
xlii	Amoa Chemical Co Ltd
xxii	Badische Anilin & Soda Fabrik A G
vi	F W Berk & Co Ltd
Cover	Bradford Dyers' Association Ltd
xx	Brotherton & Co Ltd
ii	Brown & Forth Ltd
iii	Catomance Ltd
xxv	Ciba Ltd
xxxiii	Chapman & Hall Ltd
xxvii	Clayton Dyestuffs Co Ltd
viii	Cole & Wilson Ltd
ix	Colne Vale Dye & Chemical Co Ltd
xxix	Drayton Regulator & Instrument Co Ltd
ii	Wm. Edge & Sons Ltd
xvi	Norman Evans & Rais Ltd
xl	Chas. Forth & Son Ltd
xxi	Gardinol Chemical Co Ltd
xxx	Geigy Co Ltd
xxvi	Glovers Chemicals Ltd
xi	Hardman & Holden Ltd
v	Hathernware Ltd
xxiii	Hexoran Co Ltd
vii	L B Holliday & Co Ltd
iv	Hunt & Moscrop Ltd
xiv	Imperial Chemical Industries Ltd
xxviii	Imperial Chemical Industries Ltd
v	Laporte Chemicals Ltd
xlvii	John W Leitch & Co Ltd
xl	H K Lewis & Co Ltd
xxxvi	Longclose Engineering Co Ltd
xliii	Mather & Platt Ltd
xli	Newell (Chemicals) Ltd
xliv	Samuel Pegg & Son Ltd
ii	L J Pointing & Son Ltd
xix	James Robinson & Co Ltd
xiii	Sandoz Products Ltd
xv	Sandoz Products Ltd
xxxii	Sandoz Products Ltd
viii	T Saville Whittle Ltd
i	Shell Chemical Co Ltd
ix	Shirley Developments Ltd
xxiv	F Smith & Co (Whitworth) Ltd
xxxviii	Standard Chemical Co Ltd
xlii	Staveley Iron & Chemical Co Ltd
xxxv	Tennants Textile Colours Ltd
x	W P Thompson & Co
xxxiv	Town End Chemical Works Ltd
xli	Tragasol Products Ltd
ii	J B Wilkinson (Chemicals) Ltd
vii	Williams (Hounslow) Ltd
xviii	Yorkshire Dyeware & Chemical Co Ltd

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